REVISED SAMPLING AND ANALYSIS PLAN
MACROINVERTEBRATE STUDY AND FISH SAMPLING

REMEDIAL INVESTIGATION (RI)/FEASIBILITY STUDY (FS)

McINTOSH PLANT SITE
OLIN CORPORATION
McINTOSH, ALABAMA

Prepared for
Olin Corporation
Charleston, Tennessee

October 1991

WCC File 90B449C-3A

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October 29, 1991

VIA FEDERAL EXPRESS

Kenneth A. Lucas
Senior Remedial Project Manager
United States Environmental Protection Agency
345 Courtland Street Northeast
Atlanta, Georgia 30365

Re: Revised Sampling and Analysis Plan
Olin Chemicals/McIntosh Plant Site
McIntosh, Alabama

Dear Mr. Lucas:

The Revised Sampling and Analysis Plan for Macroinvertebrate Study and Fish Sampling is enclosed. This Plan combines and incorporates EPA's comments on both the Revised Sampling and Analysis Plan for Macroinvertebrate Study and Fish Sampling submitted September 5, 1991, and the Fish Sampling Analytical Techniques submitted September 26, 1991. EPA's comments were conveyed to Olin in your letters of October 15, 1991, and October 24, 1991, and at our meeting in Atlanta of October 7, 1991. The revised Plan reflects commitments made by Olin at the meeting and in our letters of October 10 and October 18, 1991.

Woodward-Clyde will begin the sampling under this Plan on November 4, 1991. The change in schedule from the originally planned October 7 has resulted in the need for flexibility in which Woodward-Clyde crew will be involved. As a result, the exact sampling equipment may differ from the detailed description in the September submissions. The text of this Plan reflects a more general description. The equipment used will in all cases be equivalent.

As noted above, we have incorporated the changes per your October 24, 1991, letter. The "filet plus remains" method to obtain a whole-body concentration in fish is a sound method that results in more data from a given collection effort. We request that EPA provide it's rationale for not accepting this method. We also have concerns

about changes in an approved Work Plan which we will document in separate correspondence.

Please let me know if you have any questions regarding the contents of this submission or any of the work in progress at McIntosh, Alabama.

Sincerely,

OLIN CORPORATION

J. C. Brown
Manager, Environmental Technology

\jcb
Enclosure

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TABLE OF CONTENTS

TABLE OF CONTENTS

TABLE OF CONTENTS (Continued)

TABLE OF CONTENTS (Continued)

LIST OF APPENDICES

1.0
INTRODUCTION

The Olin Chemicals McIntosh plant is located approximately one mile east-southeast of the town of McIntosh, in Washington County, Alabama. A site location map is presented in Figure 1. The property is bounded on the east by the Tombigbee River, on the west by land west of U. S. Highway 43, on the north by the Ciba-Geigy Corporation plant site and on the south by River Road.

Olin operated a mercury cell chlorine-caustic soda plant on a portion of the site from 1952 through December 1982. In 1954, Olin began construction of a pentachloro-nitrobenzene (PCNB) plant on an adjacent portion of the site. The plant was completed and PCNB production was started in 1956. The McIntosh plant was expanded in 1973 to produce trichloroacetonitrile (TCAN) and 5-ethoxy-3-trichloromethyl-1,2,4-thiadiazole (Terrazole). The PCNB, TCAN and Terrazole manufacturing areas were collectively referred to as the Crop Protection Chemicals (CPC) plant. In 1978, Olin constructed a diaphragm cell caustic soda/chlorine plant which is still in operation. The CPC plant and mercury cell plant were shut down in late 1982. The McIntosh plant continues to operate and produce chlorine, caustic soda, sodium hypochlorite, sodium chloride and blends hydrazine.

The Olin McIntosh plant currently monitors and reports on numerous facilities permitted through the U.S Environmental Protection Agency (EPA) and the Alabama Department of Environmental Management (ADEM). These include water and air permits as well as a Resource Conservation and Recovery Act (RCRA) post-closure permit (including a groundwater corrective action pumping/treatment program), Solid Waste Management Unit (SWMU) closures, three injection wells for mining salt and a neutralization/percolation field.

In September 1984, Olin's McIntosh plant site was placed on the National Priority List of the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) or "Superfund". Groundwater contamination at the site has been established

based on the results of various investigations. Mercury and chloroform are the principal contaminants identified at the site. Mercury contamination was evidently caused by the operation of the mercury cell chlor-alkali plant during the period 1952 to 1982. The chloroform contamination is probably a degradation product from the operation of the CPC plant from 1954 to 1982.

Investigations have also indicated contamination in a 65-acre natural basin, herein referred to as the "basin," located on the Olin property east of the active plant facilities. This basin received plant wastewater discharge from 1952 to 1974.

In November 1989, Olin submitted a Remedial Investigation/Risk Assessment (RI/RA) report to EPA for the McIntosh facility (ERM, 1989). This report was prepared to present all relevant data, interpretations, findings and conclusions arising from previous groundwater and soils investigations, the ongoing RCRA groundwater corrective action program, and other environmental investigations that have been conducted by Olin at the McIntosh facility.

On December 5, 1989, representatives of the EPA met with Olin representatives to discuss the McIntosh site, including the adequacy of Olin's previously submitted RI/RA report (ERM, 1989) and an Administrative Order by Consent for Olin to conduct further work. The EPA indicated it would determine the adequacy based on: 1) a review of the data to ascertain completeness and 2) a determination as to whether the appropriate Quality Assurance/Quality Control (QA/QC) protocols were met during the collection and analysis of the data presented in the RI/RA report.

On January 15, 1990, Olin received a special notice letter from the EPA announcing EPA's intent to conduct an RI/FS beginning in June 1990. Olin was invited to respond to EPA with a Remedial Investigation and Feasibility Studies (RI/FS) Scope of Work to provide a basis for further negotiations. Olin submitted a draft Scope of Work to EPA on March 15, 1990. On April 9 and April 10, 1990, Olin and its consultants met with EPA Region IV representatives in Atlanta and discussed this draft. In response to comments by EPA, Olin revised the Scope of Work and submitted the final draft to the EPA on April 25, 1990.

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On May 2, 1990, Olin signed the Administrative Order by Consent (Consent Order) issued by EPA for the preparation, performance and oversight costs for the RI/FS at the McIntosh plant site. The final Scope of Work was attached to the Consent Order, which became effective May 9, 1990. A Sampling and Analysis Plan (SAP) was developed in partial fulfillment of the work items to be performed under the jurisdiction of the Consent Order. The SAP was submitted to EPA with the Amended Work Plan on May 25, 1991. This revised SAP is being submitted to supplement the original SAP by providing details regarding the biological sampling (macroinvertebrate survey and fish sampling procedures).

Two operable units have been designated for the facility. Operable Unit 1 (OU-1) is the RCRA plant area (all of the Olin property except the area defined as OU-2). Operable Unit 2 (OU-2) is the basin, including the wetlands within the Olin property line and the wastewater ditch leading to the basin. Figure 2 is a facility layout map delineating the boundaries of the two operable units.

2.0
SITE BACKGROUND AND SETTING

The information on the site background and setting presented in this section has been obtained from the ERM Remedial Investigation/Risk Assessment (RI/RA) report previously submitted to EPA.

The regional setting for the site is the East Gulf Coastal Plain Physiographic Province. Specifically, the 1500 acres that comprise the Olin property are within the Southern Pine Hills District.

SITE DESCRIPTION

The Olin McIntosh plant is an active chemical production facility. The main plant and associated Olin properties cover approximately 1,500 acres, with active plant production areas occupying approximately 60 acres. Current active facilities at the plant include: a diaphragm cell chlorine and caustic production process area; a caustic concentration process area; a caustic plant salt process area; a hydrazine blending process area, shipping and transport facilities; process water storage, transport and treatment facilities; and support and office areas. Beyond the active production facilities, the Olin property is heavily forested. A basin area is located on the Olin property, adjacent to the Tombigbee River and east of the active plant facilities.

The biological sampling that is described in this revised SAP will be conducted in OU-2. Operable Unit 2 consists of the basin (65-acres), the wetlands within the Olin property line and the wastewater ditch leading to the basin. During the seasonal high water levels (approximately 4 to 6 months per year), the basin is inundated by, and thus becomes contiguous with, the adjacent Tombigbee River.

From 1952 to 1974, plant wastewater discharge was routed through the basin and then to the Tombigbee River. In 1974, the discharge was rerouted directly to the discharge channel of the basin bypassing the basin itself. The discharge channel of the basin is

approximately 800 feet long (during the non-flood season) and flows towards the Tombigbee River. The wastewater ditch currently carries the NPDES discharge toward the Tombigbee River and stormwater runoff from the east and southeast non-manufacturing property.

In 1988, Olin completed the Basin Study Report. The study was done to provide information for the remedial investigation in accordance with CERCLA and in response to the Forward Planning Study of 1986. Sampling of both sediment and water was conducted on December 8 and 9, 1987, under the observation of EPA Region IV officials. In addition to chemical analyses, temperature and pH profiles were obtained. The analytical parameters for the basin samples included:

Water	Sediment
Total Mercury	Total Mercury
1,2-Dichlorobenzene	Soluble Mercury
1,4-Dichlorobenzene	1,2-Dichlorobenzene
Hexachlorobenzene	1,4-Dichlorobenzene
Pentachloronitrobenzene	Hexachlorobenzene
	Pentachloronitrobenzene

The mercury level detected in sediment ranged from <0.3 to 60.5 mg/kg across the basin. However, a duplicate sample analysis revealed a 9.0 mg/kg of mercury concentration corresponding to the 60.5 mg/kg value. Other sediment values ranged from 0.4 mg/kg to 25.5 mg/kg of mercury. Pentachloronitrobenzene (PCNB) was detected in three sediment samples, up to a maximum concentration of 14.5 ppm. Detection of PCNB was not confirmed in two of the three samples by replicate analysis. Hexachlorobenzene (HCB) was detected in five of the ten sediment samples. The detectable concentrations of HCB ranged from 1.9 to 114 mg/kg with a detection limit of 0.66 mg/kg. All detectable concentrations of HCB were verified with replicate analysis. The maximum concentration, 114 mg/kg, was 69.2 mg/kg in the replicate sample, thus the average value for this sample was less than 100 mg/kg (Olin, 1988).

Mercury in water was detected at or below the drinking water standards and ranged from 0.4 μg/l to 2.0 μg/l. None of the organics analyzed were detected in water.

3.0
DATA QUALITY OBJECTIVES

The biological sampling is being conducted to obtain the data necessary to evaluate the potential effects of basin contamination on the aquatic biota. The major objectives for collection of these data are as follows:

- Determine the potential presence and distribution of certain compounds in the biota.
- Support the development of the ecological assessment.
- Provide data for the Human Health Risk Assessment to assess the potential human health risk from ingestion of fish.

The biological sampling includes a macroinvertebrate survey and fish sampling.

3.1 MACROINVERTEBRATE STUDY

A study of aquatic macroinvertebrates in the basin and associated ditches will be undertaken to assess potential biological impacts. The basic information on identities and numbers of benthic macroinvertebrates observed or collected per unit of effort or unit of area will be reported as well as the resulting statistics calculated for use in comparisons.

The resultant data from the benthic macroinvertebrate identifications will be subjected to several measures to determine if differences exist among sampling sites. In addition to taxonomic lists of individual taxa and major groupings by replicate and by site, the data will be examined using CLUSTER, SIGTREE, and COMTRE index analyses. CLUSTER is a similarity index using the Bray-Curtis coefficient with unweighted average linkage and the distance linkage scale. This program groups the replicates according to both composition and abundance to determine their similarity. The data

are then subjected to SIGTREE, which determines the significance within a cluster, indicating that level of branching which constitutes a significant grouping. An hypothesis is formulated to test statistically whether two clusters within the overall cluster analysis results are sufficiently alike that they represent the same community. The third method listed is COMTRE, which compares two clusterings (also called dendrograms or trees) to determine if they are related or if the clusterings are random. These clusterings can result from the benthic macroinvertebrate data, chemical/physical data, or any of the available measurements. For example, this method can test every possible combination of the resultant benthic tree with the resultant sediment chemistry tree to determine if the benthic tree can be related to the sediment chemistry tree.

If sufficient data are available to be compared with established classifications, a biotic index, such as the North Carolina Biotic Index or the Hilsenoff Biotic Index, will be used to detect differences among sampling stations. The degree of usefulness of such indices will be dependent upon the abundance and diversity of the benthic macroinvertebrates, which are as yet unknown.

Other indices such as the EPT [percent Ephemeroptera, Plecoptera, and Trichoptera (mayflies, stoneflies, caddisflies) to the total]; percent dominance, or percent OAC (Oligochaetes, Air-breathers and Chironomidae) could be used if the taxa appearing in samples are appropriate to warrant these types of analyses.

Two sediment replicates will be taken along with each set of macroinvertebrate samples (three separate grabs in a given station) to determine particle sizes, thus helping to define/confirm comparable substrata. Results from the particle size analyses will be subjected to the SIGTREE analysis to determine if sediments from any sites are significantly different. If there is no significant difference between sediments at any site, this parameter can be eliminated as a variable in the benthic composition, abundance, and distribution analysis. If there is a difference, the results will be used to determine if sediment sizes contribute to the benthic composition, abundance, and distribution analysis. If there is a difference, the results will be used to determine if sediment sizes contribute to the benthic community structure. Additionally, one sediment sample from each macroinvertebrate location will be analyzed for Total Organic Carbon (TOC). A

Hydrolab® will be used to measure pH, temperature, specific conductance, and dissolved oxygen of the water directly above the sample locations. Depth measurements will also be obtained.

3.2 FISH SAMPLING

Tissues from fish species representing both the top of the aquatic food chain (predatory fish) as well as the bottom-dwelling, bottom-feeding species will be sampled and analyzed for mercury and selected organic contaminants. The fishes selected will include those likely to be consumed by humans and wildlife. Within these constraints, species that are most likely to be indigenous to the basin and sedentary (i.e., least migratory) will be chosen. Accordingly, largemouth bass and either yellow or black bullheads will be sampled if available in sufficient numbers. In addition to the sampled fishes, the identities and catches-per-unit-effort of fish taken during sampling for tissue will be recorded to provide information on the basin fish communities under nonflood conditions.

4.0
SAMPLE LOCATION AND FREQUENCY

Phase I sampling activities as described in the SAP have been initiated at the site. A bathymetric survey, sediment sampling and a preliminary fisheries reconnaissance were conducted as part of Phase I. Observations and preliminary results from these activities were used to develop the macroinvertebrate sample locations, the methods for the macroinvertebrate study, the fish target species, and methods for fish sampling.

4.1 MACROINVERTEBRATE STUDY

The Phase I bathymetric survey, sediment sampling and fisheries reconnaissance revealed the following information of relevance to macroinvertebrate sampling in the basin:

- Contrary to expectation, virtually none of the shallow margins of the basin exhibits emergent herbaceous aquatic vegetation; thus, the basin nearly lacks a littoral zone. During the macroinvertebrate study, observations will be made for any apparent evidence of why an extensive littoral zone is not present in the basin.

- In terms of outward appearance (color, texture, apparent grain-size composition), the upper layer of sediment is fairly uniform throughout the basin.

- Contrary to expectation, the sediments encountered during Ekman grab sampling are consistently of relatively fine particles and contain minimal coarse debris (e.g., leaves, twigs).

It thus appears that the only physical or "structural" factor likely to significantly affect the distribution of substrate associated macroinvertebrates in the basin is water depth with related factors of temperature, light, and dissolved oxygen.

Because emergent herbaceous vegetation extends for only a few meters along the shoreline in the northeast portion of the basin, studies of littoral macroinvertebrate communities will not be conducted. Instead, more effort than previously contemplated will be devoted to sampling the limnetic (open water) areas. Sampling will consist of triplicate Ekman grabs at each of 20 evenly-spaced stations selected from among the 77 basin grid nodes and two stations in the lower end of the former plant effluent channel (Figure 3). This will result in a total of 66 macroinvertebrate samples representing 22 locations throughout the entire basin and its former influent channel.

4.2 FISH SAMPLING

Twenty-two specimens of each of two species of fish will be collected from the basin. Eleven samples of each species will be prepared for whole-body analysis and eleven samples of each species will be prepared for filet analysis. In addition to the sampled fishes, the identities and catches-per-unit-effort of fish taken during sampling for tissue will be recorded to provide information on the basin fish communities under nonflood conditions. Fish that are collected but not used for tissue analysis (i.e., additional fish captured in the nets or netted during electrofishing) will be weighted and measured (total length). Fish sampling will be conducted throughout the basin until the specified numbers of specimens are obtained.

During the preliminary fisheries reconnaissance, largemouth bass were readily available, but neither bullhead species was observed. Even so, methods not tried during the reconnaissance (particularly hoopnetting and/or hook-and-line) may capture these bottom-dwelling fishes. As expected, the more migratory channel catfish (Ictalurus punctatus) was encountered during preliminary sampling. The following strategy is therefore proposed for the formal sampling effort:

- Intensive efforts will be devoted to capturing 22 specimens of either bullhead species for three days, during which time up to 22 adult channel catfish (if captured) will be accumulated and stored frozen.

- If fewer than 10 bullhead have been captured in three days, the channel catfish will be thawed and dissected for preparation as the bottom-feeding fish for analyses. (If 10 or more bullhead have been accumulated, efforts to acquire the remaining specimens will continue.)

- In the unlikely event that fewer than 10 channel catfish or bullhead have been accumulated in the first three days, the more abundant of either common carp (Cyprinus carpio) or freshwater drum (Aplodinotus grunniens) will be sought by intensive electrofishing and/or gillnetting until 20 specimens of adult bottom-feeding fish of a single species are obtained. (Note that gillnetting will be avoided until difficulty is recognized in obtaining sufficient specimens by other techniques. Gillnetting in a basin such as the basin is likely to be inordinately destructive of nontarget fishes.) Both carp and drum were readily captured during the fisheries reconnaissance.

5.0
SAMPLING EQUIPMENT AND PROCEDURES

The procedures and equipment to perform the biological sampling for RI/FS are presented herein.

5.1 MACROINVERTEBRATE STUDY

Substrate-associated aquatic macroinvertebrates will be sampled from 20 stations in the basin, and from two stations in the lower portion of the former plant effluent channel (Figure 3).

5.1.1 Equipment

The macroinvertebrate and sediment samples will be collected with a standard (6 x 6-inch) Ekman grab dredge. The samples will be sieved through a No. 60 U. S. Standard Testing Sieve (0.25-mm mesh). Water quality parameters (pH, temperature, specific conductance, dissolved oxygen and pH) will be obtained with a Hydrolab®.

5.1.2 Procedure

Water quality parameters (pH, specific conductance, temperature, and dissolved oxygen) will be obtained directly above the sediment prior to sample collection with a portable Hydrolab®. The instrument will be operated and calibrated in accordance with the manufacturers recommendations. Calibration is accomplished by immersing the sensors in standard solutions, waiting for stable readings and briefly interpreting the data printout to set new calibration points. Calibration will be conducted every time the instrument is connected to the computer or a minimum of once daily. Depth measurements will also be taken at each location.

One sediment sample will be collected at each macroinvertebrate sample location for TOC analysis. The sediment samples will be placed in 8-oz/250 ml amber, wide-mouth

jars with teflon screw caps and immediately placed in coolers with ice. The sediment samples will be shipped to the laboratory in these coolers under established chain-of-custody procedures.

At each station, three 0.023-m^2 grab samples will be collected and processed separately. The contents of each grab will be rinsed and concentrated through a No. 60 U. S. Standard Testing Sieve (0.25-mm mesh). Materials retained by the sieve will be transferred to an appropriate-sized, prelabeled container and preserved in 5 percent formalin for transport to the laboratory. The preservative will be buffered and will contain an animal tissue-specific stain (Phloxine-B) to facilitate later separation from extraneous material.

Based on observations made during Phase I, it appears that most, if not all, of the sediment particles collected in each Ekman grab will easily pass through the No. 60 sieve, leaving only macroinvertebrate animals and limited quantities of organic detritus. In the laboratory, each sample will be washed in a No. 60 sieve to remove any remaining fine sediment particles and the formalin. After the macroinvertebrates are stained and washed, they will be stored in 40 percent isopropyl alcohol if they are not processed immediately. Materials retained by the sieve will be examined under magnification and the macroinvertebrates will be separated from detrital material and sorted into major taxonomic categories (e.g., amphipods, insect orders, oligochaete worms). All specimens in each category will be counted, and up to the first 200 individuals will be identified to the lowest positive and practical taxonomic level, thus providing a basis for calculating overall numbers (by category) per unit of sampling effort and for summarizing the qualitative composition of each sample. By applying this technique to each of the three grabs from a station, the likelihood of failure to document the occurrence of any ecologically significant members of a category is very remote. A partial listing the taxonomic references that will be used is presented in Appendix A.

QA/QC practices for laboratory analyses of the benthic invertebrates are:

- After sorting, identification, and counting, 20 percent of the samples (i.e., 13 of the 66) will be redone by a qualified individual other than the person who performed the initial processing.

- Representative specimens for each of the taxa identified in 20 percent of the samples (i.e., 13 of the 66) are submitted to outside taxonomic experts for verification of identifications.

- For any taxa where doubt exists as to the accurate identification, representative specimens are sent to outside taxonomic experts.

- A voucher collection will be established of representative specimens of all taxa identified during the study.

5.2 FISH SAMPLING

The intent is to collect a minimum of 22 individuals each of a predacious species representing the top of the aquatic food chain and a bottom-dwelling, bottom-feeding species. If sufficiently abundant, largemouth bass (Micropterus salmoides) and either yellow or black bullhead (Ameiurus natalis or A. melas) will be sampled to represent the predator and bottom-feeder, respectively. If not found in sufficient quantity, additional species may be substituted as described in Section 4.2. Discrete filets will be removed from 50 percent of the sampled fish. The remaining 22 fish (11 of each species) will be prepared for whole-body analysis.

During collection of the fish for tissue analyses, numbers and identities of other species captured or observed will be recorded to provide a general picture of the fish community structure of the basin. It is anticipated that DC electrofishing and hoopnetting will be the primary fish collecting methods, although other gear such as seines, gillnets, and hook-and-line may be used. Fish that are collected but not used for tissue analysis (i.e., additional fish captured in the nets or netted during electrofishing) will be weighted and measured (total length).

Aging will be conducted on the fish that are collected for tissue analysis. The aging of the fish will include the removal of otoliths and scales from the largemouth bass and the removal of the pectoral spines from the bullheads or channel catfish; the preparation of otoliths and scales (or pectoral spines) for aging; and the performance of age estimation.

In order to conduct fish sampling activities in the basin, a scientific fish collection permit has been obtained from the Alabama Department of Fish and Game.

5.2.1 Equipment

A combination of electrofishing equipment, hoop nets, gill nets, hook and line, and seines will be used for collection of fish.

5.2.2 Procedures and Equipment Used in Electrofishing

The primary fish collecting device will be the boat-mounted fish electroshocker. Two persons operate the boat-mounted electroshocker, one person to run the trolling motor and steer the boat and the other person to operate the shocker and collect the stunned fish as they float to the surface. Fish collection by electroshocking is usually conducted along the shoreline, typically within 50 feet of the bank. Electrofishing is normally performed during the early morning and the early evening hours in order to conduct sampling coincident with peak fish activity patterns and to minimize the stress on the captured fish.

The shocker is operated by the use of a foot control pedal "kill" switch. Through reactions known as electrotaxis (DC) and oscillotaxis (AC), the fish are stunned by the electrical field and float to the surface, where they are collected with a dip net and placed in a holding container. As a safety factor, while electroshocking activities are underway, occupants of the boat wear rubber-soled footwear and rubber "lineman type" gloves. Electrofishing activities are halted periodically to sort the collected fish in the holding container. Fish samples are placed in bags and held on ice in a cooler to await categorization and processing. Fish that are not used are released. The electrical field is activated only when the "kill" switch foot pedal is depressed by the operator.

5.2.3 Procedure Used for Fish Collection with Hoopnets

The hoopnets may be used primarily for the collection of catfishes. Two different sizes of hoopnets are used, one 7 feet in length and 30 inches in diameter and the other 5 feet in length and 24 inches in diameter. The hoopnets may be baited with cheese, which is placed in a chamber designed for that purpose in the closed end of each net. Any bait cheese that is used will be analyzed for the same parameters as the fish tissue. The hoopnets are set along the shore or across channels in locations which are judged to be most appropriate. Attracted by the cheese bait in the closed end of the net, fish enter the net and cannot leave. Typically, the nets are set in the evening and checked in the morning after electrofishing activities are concluded. The fish are collected when the net is pulled. The nets are then rebaited and set again to collect fish for the following day. The fish, once collected, are either put in a labeled plastic garbage bag and placed on ice in a cooler or released. The fish in the coolers are held for categorization and processing for mercury analysis and other data collection.

5.2.4 Procedures Used for Fish Collection with Gill Nets

The gill nets are of varying lengths, depths, and mesh sizes depending on the types of fish sought. The gill nets are weighted along the bottom line and buoyed along the top. The buoys are painted a fluorescent orange color so that they can be easily seen by any boat traffic. Any fish swimming into the net can become entangled in it. The gill nets are set in the late afternoon and checked daily each morning. The fish are collected as the nets are pulled. The fish collected from the nets are placed on ice in coolers for categorizing and processing. Any fish collected alive and not needed for sample analysis is immediately released.

5.2.5 Procedures Used for Fish Collection with Seines

The seine used normally for fish collection is a nylon drag seine with 1-inch mesh consisting of Number 15 nylon netting. The seine is 6 feet from top to bottom and is 25 feet long and has a mud line along the bottom which facilitates the collection of fish in shallow shoreline areas, especially in small bays and coves. Other similar seines may be used. The technique is used to catch smaller fishes and is employed primarily as a backup to other fish collection methods.

5.2.6 Fish Categorization and Field Sample Preparation

All fish collected for chemical analysis by the various procedures described above will be transported in coolers on ice from the field to a site for categorization and field sample preparation. Field sample preparation will be conducted on a hard ceramic-type cutting board covered by polyethylene plastic. A new piece of plastic will be used for each fish. Prior to any fish processing, the cutting board will be decontaminated using the procedures outlined in Section 5.4. Decontamination will also be conducted in the event the plastic tears allowing contamination of the cutting board. Each fish will be assigned a sample number, weighed, and measured (total length). Following the collection of this information, filet samples will be obtained from 50 percent of the fish collected (11 from each species) and weighed. The otoliths and scales will be removed from the largemouth bass and the pectoral spines will be removed from the bullheads or channel catfish for age determination (see Section 5.2). The filet samples that are sent to the laboratory for processing and homogenization will include the complete filet from both sides of the fish. For the bullheads or channel catfish, the filets will be processed with the skin off. The largemouth bass filets will be processed with the skin on after removing the scales. The filet samples will be placed in separate appropriately labeled glass jars with teflon-lined screw tops and immediately placed in coolers with ice. These whole body fish will be wrapped in aluminum foil (shiny side out) and immediately placed in coolers with ice. One of the prepared filet samples of each species and one whole-body specimen of each species will be provided to the EPA oversight contractor. The remaining samples (both filet and whole body) will be stored on ice and sent under established chain-of-custody procedures to the laboratory for

analysis. Further processing of the fish samples (i.e., homogenization of the samples) will be conducted at the analytical laboratory as described in Section 7.2.

5.3 SAMPLE DESIGNATION

All samples will be identified by a unique numbering system. Additional identification will consist of sample type (fish, macroinvertebrate, etc.), location (grid location number for macroinvertebrates), and analysis.

Labels will be used for sample security, identification, and integrity. Information on the sample container will include the following:

- WCC project number
- Sample station number
- Date and time of sample collection
- Designation of the sample as a grab or composite
- Type of sample (macroinvertebrate, fish, etc.)
- The name(s) of the sampler(s)
- Whether the sample is preserved or unpreserved
- The general types of analyses to be conducted
- Any other relevant comments

All of the containers for each sample set will bear the same number. The anticipated numbering system will consist of a unique identification number established as follows: sample matrix abbreviation/location designation/sequential number for that date. For example, the first discrete macroinvertebrate sample obtained from a location F1 will be numbered M-F1/01. Matrix spikes and matrix spike duplicates will be designated by the letters MS or MSD, respectively.

Once this information has been put on the sample label and the sample label affixed to the jar, the label will be covered with clear vinyl tape to protect this information. The sample identification code will be used to identify each sample in the master field log book and other field documentation logs.

5.4 FIELD DOCUMENTATION

Sampling activities will be documented in a bound field log book with consecutively numbered pages. Information recorded in the log book will include:

- WCC project name and number
- Location and sampling activity
- Purpose of sampling
- Number and approximate volume of samples taken
- Description of sampling point
- Date and time of collection
- Collector's sample identification number(s)
- Sample distribution (e. g., chemical laboratory, geotechnical laboratory, etc.)
- Sample preservation
- Field observations
- Any field measurements made, such as pH, specific conductivity or other field parameters
- Weather conditions

The documentation in the log book will be sufficient to reconstruct the sampling situation without relying on the collector's memory.

5.5 DECONTAMINATION PROCEDURES

All sampling equipment will be decontaminated before entering the site and leaving the site. In addition, equipment will be decontaminated between sample locations to prevent cross-contamination. Washwater that is collected during decontamination will be containerized for disposal/treatment by Olin.

The sediment and fish sampling equipment that comes in direct contact with the sample for chemical analysis (e.g., knives for fileting fish, etc.) will be decontaminated using the following procedures:

1. Clean with tap water and laboratory detergent using a brush if necessary to remove particulate matter and surface films.

2. Rinse thoroughly with tap water.

3. Rinse thoroughly with deionized water.

4. Rinse twice with pesticide-grade isopropanol.

5. Rinse thoroughly with organic-free water and allow to air dry as long as possible.

6. If organic-free water is not available, allow equipment to dry as long as possible. Do not rinse with deionized or distilled water.

7. Wrap with aluminum foil, if appropriate, to prevent contamination if equipment is to be stored or transported.

Solvents will not be used for cleaning of plastic items. Plastics may be used (instead of foil) to wrap equipment after cleaning if all traces of solvents have been removed. All decontamination fluids, except for tap water, must be applied using non-interfering containers and applicators. These should be made of glass, stainless steel or Teflon. Pump sprayers, because of the presence of rubber and greased or oiled leather gaskets and seals, are generally only acceptable for applying tap water.

To verify the adequacy of the decontamination, one rinsate sample from the sampling equipment will be obtained during the fish sampling. The rinsate samples will be analyzed for the same parameters as the media that was sampled with the equipment.

The macroinvertebrate samples will not be analyzed for chemical parameters; therefore, equipment decontamination will consist of washing with tap or basin water until visible sediment and residue is removed.

Personnel will wear appropriate protective clothing during decontamination as required by the Health and Safety Plan. All protective equipment (gloves, boots, etc.) will be decontaminated after use or they will be disposed of in drums, labeled, dated, and stored for ultimate disposal at an approved facility. Disposable safety equipment will be considered to be contaminated after use and will be packaged and disposed of in an approved manner.

6.0
SAMPLE HANDLING

6.1 SAMPLE CONTAINMENT AND SECURITY

Samples will be stored in a manner that will not jeopardize the representativeness of the media sampled. For samples to be analyzed for chemical parameters (i.e., the fish samples), normally this will mean freezing or storage on ice or refrigeration, in closed containers. Samples will be analyzed within the holding times stated in the analytical procedures discussed in Section 7.0.

Sample coolers will be under the direct observation of WCC personnel at all times or secured with custody seals to detect tampering. If samples are not attended, they will be kept in a secured facility. All samples will be turned over to the WCC field operations task leader or his designee at the end of the day, along with chain-of-custody forms and field documentation forms. Samples placed in the coolers will be packed with ice or ice packs upon retrieval and will be maintained at 4°C until delivery to the laboratory. Prior to shipment, a second person (other than the one packing the cooler) will verify samples, chain-of-custody and other documentation.

6.2 CHAIN-OF-CUSTODY PROCEDURES

The chain-of-custody procedures document sample possession from the time of collection to final disposition.

For the purpose of these procedures, a sample is considered in custody if it is:

- In one's actual possession
- In view, after being in physical possession
- Locked so that no one can tamper with it, after having been in physical custody
- In a secured area, restricted to authorized personnel.

A chain-of-custody form will be initiated in the field, and the original will accompany the samples with copies retained at intermediate steps. The following information will be specified for each sample on the chain-of-custody form:

1. Sequential sample number
2. Sample date
3. Sample time
4. Sample location and depth where appropriate
5. Analyses to be performed

The chain-of-custody form will be signed by the sample custodian. It will be placed in a water-tight plastic bag and taped to the underside of the lid of the cooler containing the samples designated on the form. The lid of the cooler will be securely taped shut with custody seals, using evidence tape to allow detection of any possible tampering. Upon arrival in the laboratory, samples will be received by the analytical laboratory representative. Samples contained in the shipment will be compared to the chain-of-custody form to ensure that all samples designated have been received. Sample custody within the laboratory will be maintained on internal tracking forms.

Each time responsibility for custody of the sample changes, the new custodian will sign the record and denote the date. An exception would be the commercial carrier, if used. A copy of the signed record will be made and retained by the immediately previous custodian and sent to the designated WCC personnel to allow tracking of sample possession. All changes of custody of samples must be a person-to-person change of physical possession.

Upon completion of the analysis, the custodian responsible for the analysis will complete the chain-of-custody record, file a copy, and send the original with results to the WCC Project Manager for record keeping.

7.0
SAMPLE ANALYSIS

The sample analyses, quality control and reporting for the target parameters in fish are similar to those discussed previously for soil samples in Sampling and Analysis Plan Quality Assurance Project Plan (QAPP) (Volume II of II, Remedial Investigation (RI)/Feasibility Study (FS), McIntosh Plant Site, Olin Corporation, McIntosh, Alabama, May 1991). The fish analytical parameters and techniques are included in this document along with revisions and additions to the May 1991 QAPP that pertain to the fish sampling.

7.1 SAMPLE PARAMETERS

The sediments collected at each macroinvertebrate location will be analyzed for Total Organic Carbon (TOC). The fish tissue will be analyzed for mercury and additional parameters based on the results of the sediment analyses. The preliminary data from the sediment analyses have been reviewed for the purpose of selecting the appropriate parameters.

7.1.1 Selection of Organic Parameters

Results of the organic analyses have been received from 15 sediment samples and one duplicate from the basin and two sediment samples from the former wastewater ditch. Figure 3 shows the sample locations. The laboratory reported data are presented in Appendix B. A summary is presented in Table 1. The criteria for selection of the organic parameters was as follows:

- Constituents most common to the basin based on the preliminary sediment data.

- Constituents from Olin most likely to be found and most likely to persist in the basin sediments based on the knowledge of past operations at the facility.

Volatile Organics

The following summarizes the selection of organic parameters based on these criteria and the results of the organic analyses. The summary refers to concentrations detected above the Contract Required Quantitation Limit (CRQL).

Parameters found in blanks were not selected for analysis (i.e., methylene chloride, acetone).

- The compound 2-Butanone was not selected because it was reported at relatively low concentrations and at these concentrations is considered a laboratory artifact.
- Carbon disulfide was not selected because it also was reported at low concentrations indicating that it is a laboratory artifact or possibly naturally occurring.
- Chlorobenzene was selected for analysis.

Semivolatile Organics

Table 1 shows the three semivolatile organic parameters that were reported above the CRQL.

- Bis(2-Ethylhexyl)phthalate was not selected. It was only found in two of 17 samples. This parameter is not believed to be indicative of organics in the basin. Phthalate esters are used extensively as plasticizers and commonly show up in environmental analyses resulting from either field or laboratory contaminants.
- Hexachlorobenzene was selected based on the sediment data. Additional chlorinated benzenes (including 1,4-dichlorobenzene) were

selected because they are known to have been present at the Olin facility when wastewater was discharged to the basin. They were selected for the fish analyses due to the potential for bioaccumulation in the fish tissue.

Pesticides/PCBs

Seventeen pesticide compounds were reported in the sediment samples. The five most commonly reported, at the highest concentrations are presented in Table 1. The remaining twelve constituents were reported at a maximum concentration of 0.11 mg/kg. None of these pesticide compounds were handled at the Olin facility.

- 4,4'-DDD, 4,4'-DDE and 4,4'-DDT were selected because these are the most commonly reported constituents in the basin (reported in all 17 samples), and these were the only pesticide compounds confirmed by Gas Chromatograph/Mass Spectrometry (GC/MS).
- Considering that the source of these pesticide compounds is not believed to be the Olin facility, additional Pesticide/PCB parameters were not selected for the fish analyses. The mobility of the fish would limit any interpretation of these data because sampled fishes may have been exposed to these non-Olin parameters at higher concentrations outside of the basin.

7.1.2 Review of Preliminary Inorganic Sediment Sample Results

Preliminary results of inorganic analyses of sediment samples have been obtained for the 17 samples and one duplicate at the locations shown in Figure 4. These results are presented in Appendix B. Mercury has been selected for inorganic analyses of the fish tissue. From knowledge of past operations at the facility and previous sediment analyses, mercury has been identified as a contaminant of concern. The results of other inorganic analyses were reviewed to assess whether these additional inorganic are present at high enough concentrations in the sediment to require analyses of fish tissue to adequately characterize the potential risk to human and biota receptors.

The inorganic constituents are found as naturally occurring at varying concentrations. Therefore, the basin sediment data are compared to reported common ranges for naturally occurring concentrations in Table 2. It should be noted that these are common ranges and values above or below the common range can be naturally occurring.

- Antimony was reported above the common range for naturally occurring concentrations in five samples at a maximum concentration of 24.6 mg/kg. The listed common range for antimony is 2 mg/kg to 10 mg/kg.

- Cadmium was reported slightly above the common range for naturally occurring concentrations in four samples at a maximum concentration of 1.0 mg/kg. The listed common range for antimony is 0.01 mg/kg to 0.7 mg/kg. The reported concentrations were also only slightly above the detection limit of 0.7 mg/kg.

- Selenium was reported slightly above the common range for naturally occurring concentrations in one sample at a maximum concentration of 2.4 mg/kg. The listed common range for selenium is 0.1 mg/kg to 2.0 mg/kg.

- Cyanide which does not have a common range listed in Table 2 was reported in 4 of the 17 samples and the duplicate sample DSG-D6 (cyanide was not found in the original sample SG-D6). The maximum cyanide concentration was 0.47 mg/kg, less, less than two times the detection limit of 0.25 mg/kg.

- Mercury was reported above the common range for naturally occurring concentrations in all 17 samples and the duplicate sample at a maximum concentration of 30.1 mg/kg. The common range listed for mercury is 0.01 to 0.3 mg/kg.

It is not evident from the data whether the reported values for antimony, cadmium, selenium, or cyanide are due to factors other than contamination. The concentrations could be due to naturally occurring variations in sediments or in the case of the values reported near the detection limits, false positives. A more thorough review of the data will be conducted during the data validation process.

The potential impacts of antimony, cadmium, selenium, and cyanide at the concentrations reported in the basin sediments as compared to mercury are relatively insignificant. Mercury is more toxic at low concentrations. Mercury is a known contaminant of concern. Given the low concentrations, the occurrence of cyanide, antimony and cadmium as contaminants is questionable. Therefore, analysis of fish for mercury will adequately characterize the potential risk of inorganic constituents to human and biota receptors, without analysis for other inorganic parameters.

7.1.3 Summary

The analytical parameters selected for the fish analyses are as follows:

Category/Parameter	CLP Target Parameter
Metals	
Mercury	Yes
Volatile Chlorinated Benzenes	
Chlorobenzene (monochlorobenzene)	Yes
Semivolatile Chlorinated Benzenes	
1,2-Dichlorobenzene	Yes
1,3-Dichlorobenzene	Yes
1,4-Dichlorobenzene	Yes
1,2,4-Trichlorobenzene	Yes
Pentachlorobenzene	Yes
Hexachlorobenzene	Yes
Pentachloronitrobenzene	No[1]
Chlorinated Pesticides	
4,4'-DDT	Yes
4,4'-DDD	Yes
4,4'-DDE	Yes
Lipids	No[1]

[1] These analyses will be conducted, but they are not CLP parameters.

7.2 ANALYTICAL METHODS

Tissue sample preparation will be performed by Hazleton Environmental Services (Hazleton) located in Madison, Wisconsin. The preparation techniques are outlined in Appendix C, Preparation of Fish Tissue for Analytical Determinations in the Laboratory, OP-6004-FPREP, Hazleton Environmental Services, Madison, Wisconsin, April 1991.

After completion of the initial fish tissue processing, the following subsamples will be weighed:

Category/Parameter	Sample Weight (grams)	Number of Aliquots
Mercury (Microwave Digestion)	1.0	3
Chlorobenzene	5.0	3
Chlorinated Benzenes, Chlorinated Pesticides, and Lipids	30	3

The sample aliquots will be stored in glass containers with Teflon-lined screw tops. Three subsamples for mercury analysis will be frozen on dry ice and shipped to Olin Chemicals laboratory in Charleston Tennessee for mercury analysis.

The remaining subsamples and bulk sample will be retained by Hazleton. The three subsamples for each category will provide one subsample for the primary analysis and two subsamples held frozen in reserve should reanalysis be required.

The fish tissue analyses will be performed using the following methods.

- **Mercury** (Presented in Appendix D)
  Determination of Total Mercury in Fish, Olin Corporation Product Quality and Environmental Control Department, Charleston, Tennessee, undated.

- **Volatile Chlorinated Benzene** (Presented in Appendix E)
  Method Summary for Chlorobenzene in Fish Tissue by GC/MS, Hazleton Environmental Services, Madison, Wisconsin, September 1991.

- **Semivolatile Chlorinated Benzenes, Chlorinated Pesticides and Lipids** (Presented in Appendix F)
  Semivolatile Analysis of Fish Samples by Gas Chromatography/Mass Spectroscopy (GC/MS), MP-HZB-MA, Hazleton Environmental Services, Madison, Wisconsin, undated.

The sediment samples will be analyzed for TOC by EPA Method 9060 as described in the following reference: Test Methods for Evaluating Solid Waste - Physical/Chemical Methods.

Third Edition, EPA SW-846, U. S. Government Printing Office (955-001-00000-1), Washington, D. C., 1986.

7.3 DETECTION LIMITS

The method detection limits for the analytical parameters are as follows:

Category/Parameter	Method Detection Limit (mg/kg)
Metals	
Mercury	0.10
Volatile Chlorinated Benzenes	
Chlorobenzene (monochlorobenzene)	0.005
Semivolatile Chlorinated Benzenes	
1,2-Dichlorobenzene	0.660
1,3-Dichlorobenzene	0.660
1,4-Dichlorobenzene	0.660
1,2,4-Trichlorobenzene	0.660
Pentachlorobenzene	0.660
Hexachlorobenzene	0.660
Pentachloronitrobenzene	0.660
Chlorinated Pesticides	
4,4'-DDT	0.660
4,4'-DDD	0.660
4,4'-DDE	0.660
Lipids	Not Applicable
TOC (Sediment)	50

Elevated detection limits are anticipated only for those samples exhibiting high analyte concentration levels and/or interferences.

7.4 SAMPLE PRESERVATION AND HOLDING TIMES

Sediment samples will be placed in 8-oz/250 ml amber, wide-mouth jars. As stated in Section 5.2.6, filet samples will be placed in glass jars with teflon-lined screw tops and the whole-body samples will be wrapped in aluminum foil (shiny side out). Samples will be shipped from the field to the laboratory protected from light and at wet ice temperature. The sediment samples will be refrigerated at approximately 4° C and protected light. The fish samples will be stored, protected from light and frozen at approximately -20°C from the time of receipt in the laboratory until sample preparation and analysis.

There are no established holding times for the target analytes in fish. The analysis for chlorobenzene will be performed within seven days of sample preparation. The sediment analyses for TOC will be performed within 28 days of collection.

7.5 QUALITY CONTROL

The quality control analyses will include method blanks, duplicates and matrix spikes. The quality control analyses for the chlorinated benzenes will also include internal standards and surrogates.

Field duplicates for the TOC samples will be collected at a frequency of 1 per 20 or fewer samples.

A method blank will be processed each time 20 or fewer samples are prepared or a new source of reagent is utilized.

A laboratory duplicate and matrix spike will be analyzed for each group of 10 or fewer mercury field samples. The control limit for the matrix spike recovery is 70 to 130 percent.

A laboratory matrix spike and matrix spike duplicate will be analyzed for each group of 20 field samples for chlorobenzene, the semivolatile chlorinated benzenes and chlorinated pesticides. The control limits for the chlorobenzene will correspond to those listed for

chlorobenzene in soil in the May 1991 QAPP. The control limits for the chlorinated benzenes and chlorinated pesticides will be as follows:

Parameter	Percent Recovery	Relative Percent Difference
1,2-Dichlorobenzene	10-90[1]	49[1]
1,3-Dichlorobenzene	10-90[1]	49[1]
1,4-Dichlorobenzene	10-90	49
1,2,4-Trichlorobenzene	10-120	33
Pentachlorobenzene	10-120[1]	33[1]
Hexachlorobenzene	10-120[1]	33[1]
Pentachloronitrobenzene	10-120[1]	33[1]
4,4'-DDT	10-120[1]	49[1]
4,4'-DDD	10-120[1]	49[1]
4,4'-DDE	10-120[1]	49[1]

[1] Estimated.

A laboratory duplicate and matrix spike will be analyzed for each group of 20 for fewer TOC sediment samples.

The control limits for the TOC sediment analyses will be as follows:

- Percent recovery 75 to 125 percent
- Precision ±20 percent
- Completeness 90 percent

7.6 REPORTING

The final analytical reports will include the following:

- Case Narrative
- Chain-of-Custody
- Quality Control Summary
- Sample Data
- Calibration
- Raw Quality Control Data
- Sample Preparation Logs
- Instrument Run Logs

7.7 DATA VALIDATION

Data validation will be performed in a manner similar to that used for the Contract Laboratory (CLP) data as described in Sample Analysis Plan Quality Assurance Project Plan (QAPP) (Volume II of II, Remedial Investigation (RI/Feasibility Study (FS), McIntosh Plant Site, Olin Corporation, McIntosh, Alabama, May 1991).

TABLES

TABLE 1

SUMMARY OF PRELIMINARY ORGANIC SEDIMENT DATA[1]

Concentrations in µg/kg[2]

Sample Location	G3[3]	H4	K4	C5	C6	D6	J6	F7	J7	G8	H8	G9	C10	D10	I10	BD05	BD06
VOLATILES																	
2 Butanone	--[4]	--	--	--	64	--	--	72	62	50	--	45	200	140	60	34	--
Carbon Disulfide	--	--	--	--	--	--	--	64	--	--	--	--	--	56	--	--	--
Chlorobenzene	1000	100	--	460	380	650	--	70	--	--	--	--	48	--	--	42	46
SEMIVOLATILES																	
1,4-Dichlorobenzene	--	--	--	470	--	--	--	--	--	--	--	--	--	--	--	--	--
Hexachlorobenzene	40000	--	--	20000	--	--	--	--	--	--	--	--	--	--	1800	--	4500
Bis(2-ethylhexyl)phthalate	--	--	--	2700	--	--	--	--	--	--	--	--	--	37000	--	--	--
PESTICIDES/PCBs																	
4'4'-DDD	570	490	1800	120	760	1300	200	250	510	570	580	450	820	710	560	300	110
4'4'-DDE	930	740	1100	100	530	850	210	380	830	530	560	410	760	520	1400	260	150
4'4'-DDT	170	420	4000	52	170	290	84	79	310	170	220	190	--	360	150	170	--
Delta BHC	77	--	--	--	--	170	--	87	130	54	--	33	52	30	--	22	24
Endosulfan I	47	61	220	110	60	140	17	42	69	45	54	29	92	64	150	--	--

NOTES:

1 Only selected constituents reported above the Contract Required Quantitation Limit are listed. A complete summary with all data qualifiers is shown in Appendix B tables.

2 The listed result is either from original extract or a diluted extract. See attached tables for both results. The data remain to be validated by functional guidelines.

3 Sample location - see attached Figure 4. All sample designations were preceded by SG.

4 Either the sample was not detected or was estimated at a concentration below the CRQL.

TABLE 2

SUMMARY OF PRELIMINARY INORGANIC SEDIMENT DATA[1]

Compound	Maximum Concentration (mg/kg)	Common Range[2] (mg/kg)	Number of Sampling Outside Common Range	Detection Limit (mg/kg)
Antimony	24.6	2 - 10	5	8.0
Arsenic	12.8	1 - 50	0	1.3
Beryllium	2.5	0.1 - 40	0	0.7
Cadmium	1.0	0.01 - 0.7	4	0.7
Chromium	54.5	1 - 1,000	0	1.33
Copper	50.4	2 - 100	0	3.33
Cyanide	0.47	NA[3]	4[4]	0.25
Lead	44.2	1 - 200	0	0.4
Mercury	30.1	0.01 - 0.3	18	0.1
Nickel	25.3	5 - 500	0	5.33
Selenium	2.4	0.1 - 2.0	1	0.7
Silver	1.36	0.01 - 5	0	1.33
Thallium	0.9	NA[3]	NA[3]	0.7
Zinc	205	10 - 300	0	2.7

NOTES:

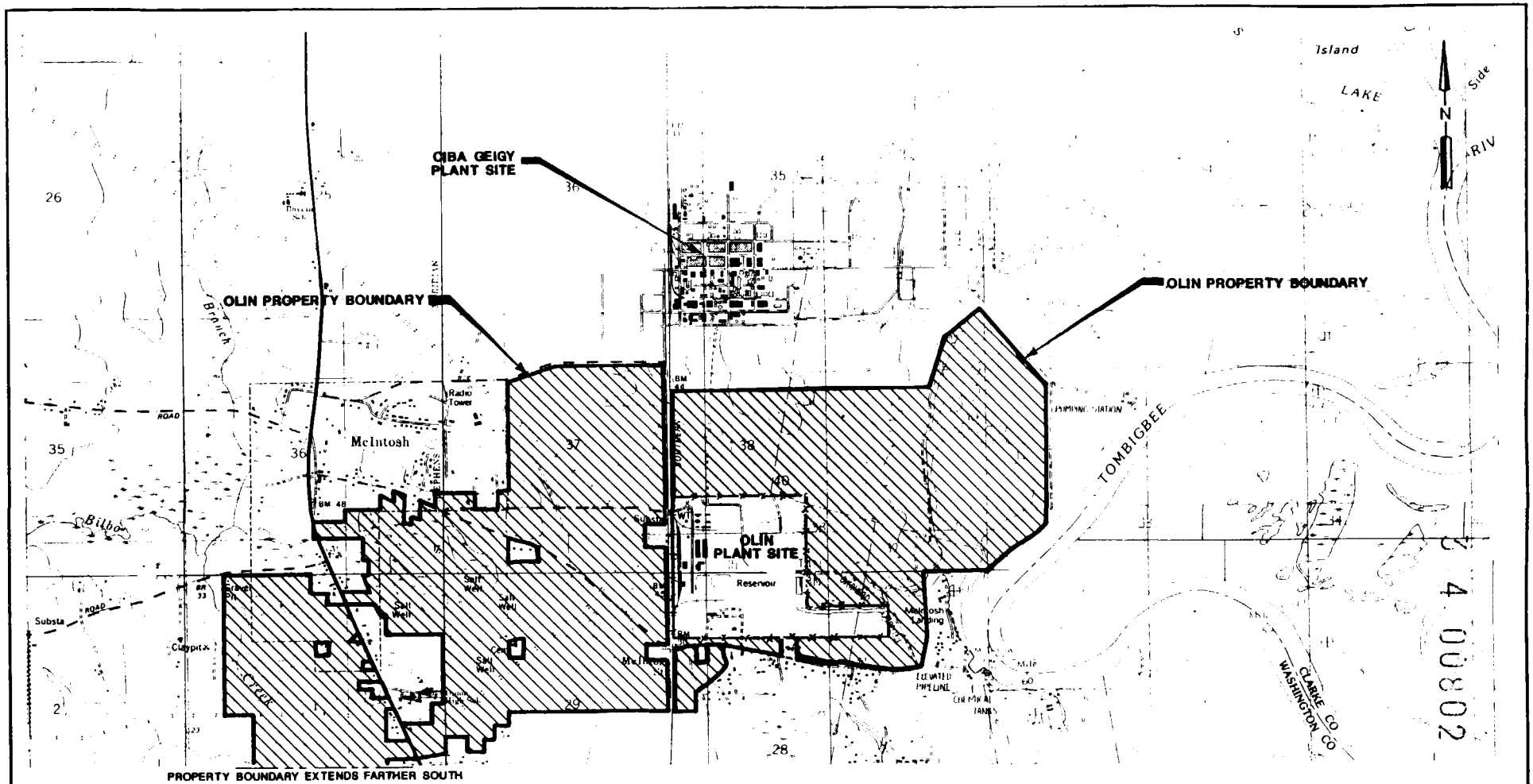
1 A complete list of results is presented in Appendix B.

2 Reference USEPA Office of Solid Waste and Emergency Response, Hazardous Waste Land Treatment, SW874 (April 1983), page 2-3, Table 6.46.

3 Common range not reported.

4 Listed as number of samples above the detection limit.

FIGURES



LEGEND

—X— OLIN PLANT SITE FENCE LINE

OLIN PROPERTY BEYOND PLANT SITE

ALABAMA

QUADRANGLE LOCATION

0 (FEET) 2000

SCALE

RE: U.S.G.S. 7.5 MINUTE SERIES QUADRANGLE MAP, McINTOSH, ALABAMA, 1964 AND GINHOUSE ISLAND, ALABAMA, 1964.

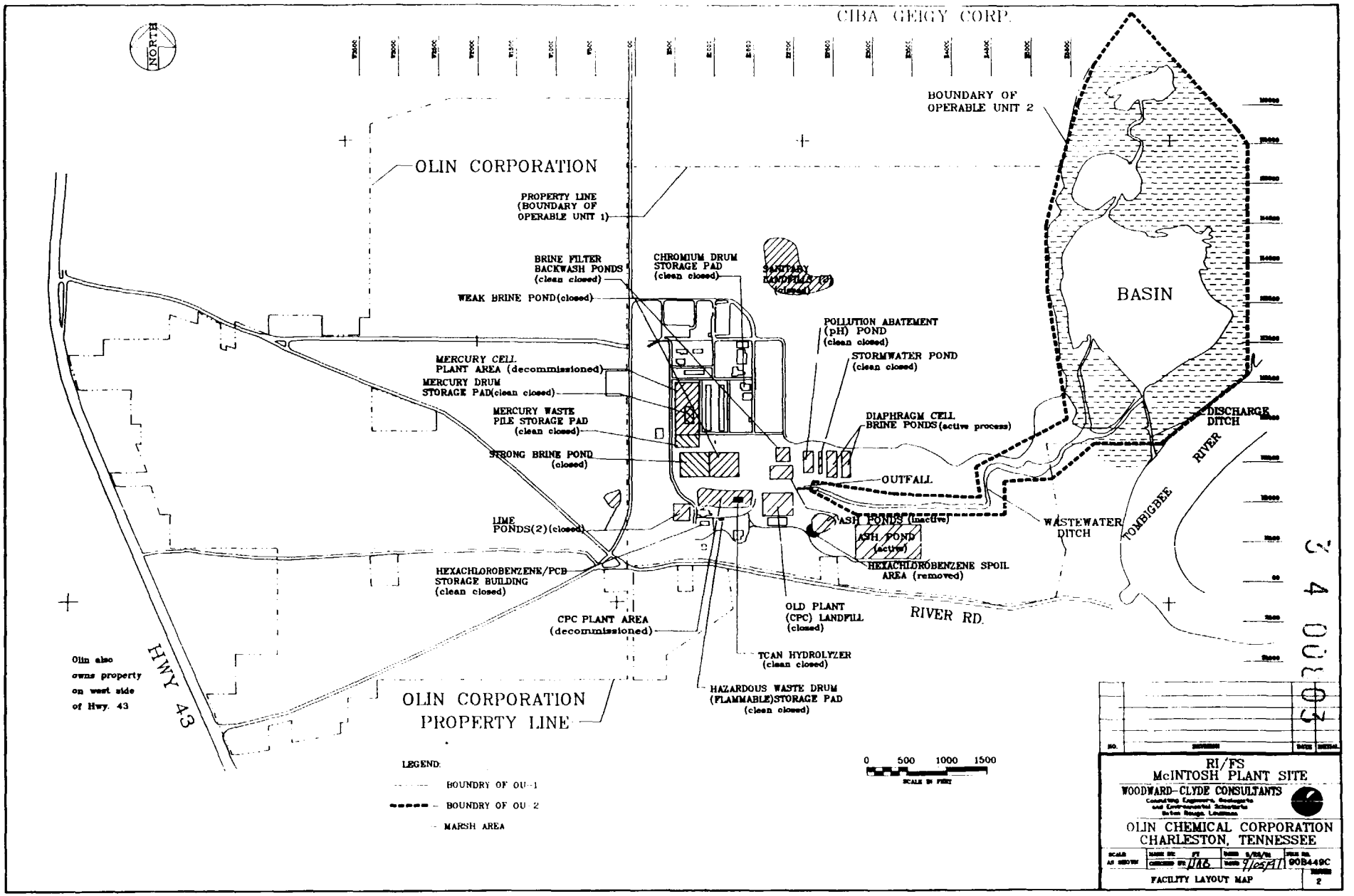
RI/FS
McINTOSH PLANT SITE

Woodward-Clyde Consultants
Consulting Engineers, Geologists and Environmental Scientists
Baton Rouge, Louisiana

OLIN CHEMICAL CORPORATION
CHARLESTON, TENNESSEE

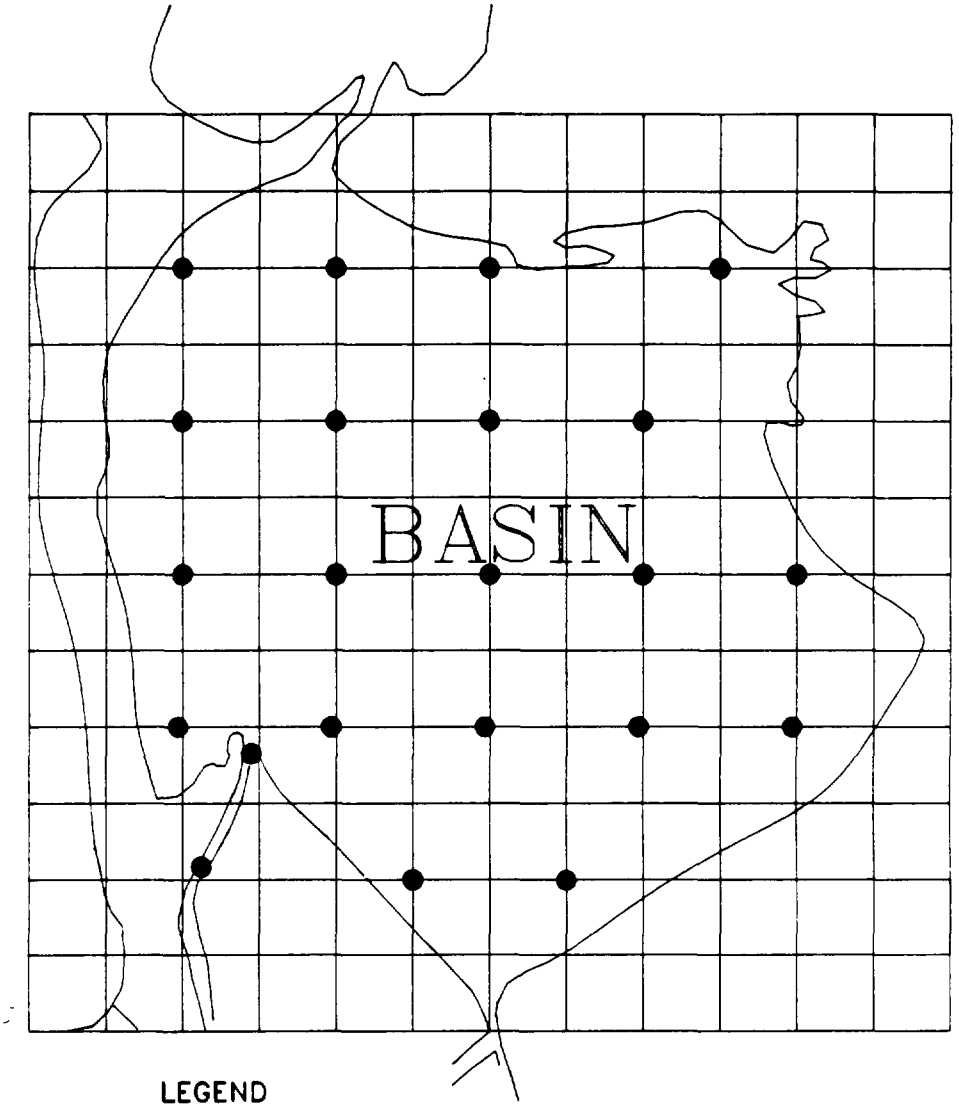
SCALE:	MADE BY: SJL	DATE: 12/90	FILE NO.
AS SHOWN	CHECKED BY: WAL	DATE: 12/9-	908449C

SITE LOCATION MAP — FIGURE 1


CIBA GEIGY CORP.
NORTH
OLIN CORPORATION
PROPERTY LINE (BOUNDARY OF OPERABLE UNIT 1)
BOUNDARY OF OPERABLE UNIT 2
BRINE FILTER BACKWASH PONDS (clean closed)
CHROMIUM DRUM STORAGE PAD (clean closed)
SANITARY LANDFILLS (2) (closed)
WEAK BRINE POND(closed)
BASIN
POLLUTION ABATEMENT (pH) POND (clean closed)
STORMWATER POND (clean closed)
MERCURY CELL PLANT AREA (decommissioned)
MERCURY DRUM STORAGE PAD(clean closed)
MERCURY WASTE PILE STORAGE PAD (clean closed)
DIAPHRAGM CELL BRINE PONDS(active process)
DISCHARGE DITCH
RIVER
STRONG BRINE POND (closed)
OUTFALL
LIME PONDS(2)(closed)
ASH PONDS (inactive)
ASH POND (active)
WASTEWATER DITCH
TOMBIGBEE
HEXACHLOROBENZENE/PCB STORAGE BUILDING (clean closed)
HEXACHLOROBENZENE SPOIL AREA (removed)
CPC PLANT AREA (decommissioned)
OLD PLANT (CPC) LANDFILL (closed)
RIVER RD.
TCAN HYDROLYZER (clean closed)
HAZARDOUS WASTE DRUM (FLAMMABLE)STORAGE PAD (clean closed)
Olin also owns property on west side of Hwy. 43
HWY 43
OLIN CORPORATION PROPERTY LINE
LEGEND:
BOUNDRY OF OU-1
BOUNDRY OF OU-2
MARSH AREA
0 500 1000 1500
SCALE IN FEET
RI/FS
McINTOSH PLANT SITE
WOODWARD-CLYDE CONSULTANTS
OLIN CHEMICAL CORPORATION
CHARLESTON, TENNESSEE
90B449C
FACILITY LAYOUT MAP
2

S4 00103

3 4 00804



LEGEND

● – MACROINVERTEBRATE SAMPLE LOCATION

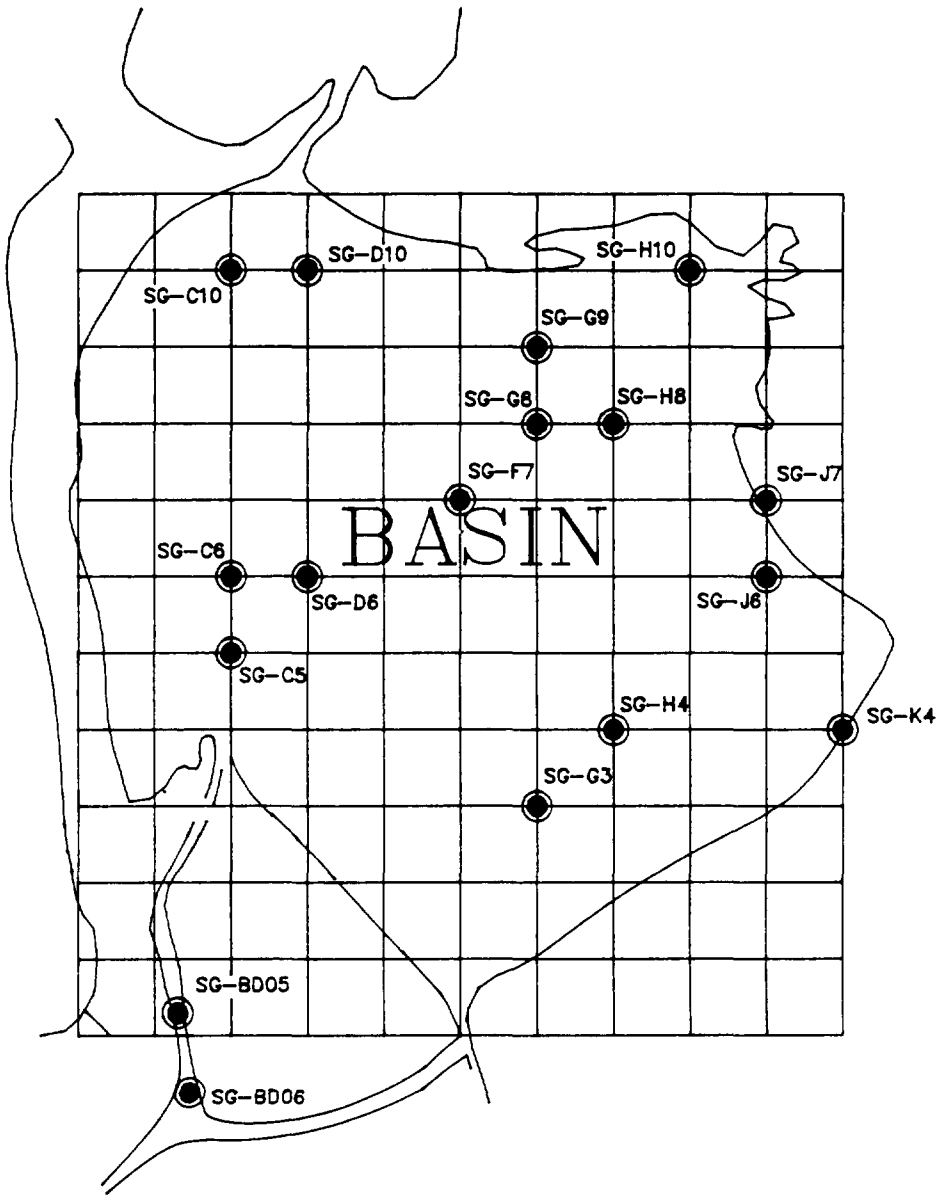
SCALE

500 0 500 1000 FEET

NOTE: LOCATIONS ARE APPROXIMATE.

RI/FS McINTOSH PLANT SITE OLIN CHEMICAL CORP. CHARLESTON, TENNESSEE	**Woodward-Clyde Consultants** Consulting Engineers, Geologists and Environmental Scientists Baton Rouge, Louisiana	MACROINVERTEBRATE SAMPLE LOCATIONS	FILE NO. 90B449C
	SCALE: AS SHOWN; DRAWN BY: J. BICKFORD; DATE: 8/27/91; CHKD. BY: WAC; DATE: 5/29/91		FIG. NO. 3

3 4 00805



LEGEND

● CLP GRAB SAMPLE LOCATION

SCALE

500 0 500 1000 FEET

NOTE: LOCATIONS ARE APPROXIMATE.

Location: B.R.,LA File name: K:\DRWG\OLIN\0B449C1A.DWG Last edited: 10/24/91 ● 10:03

RI/FS McINTOSH PLANT SITE OLIN CHEMICAL CORP. CHARLESTON, TENNESSEE	**Woodward-Clyde Consultants** Consulting Engineers, Geologists and Environmental Scientists Baton Rouge, Louisiana	*BASIN GRAB CLP SEDIMENT SAMPLE LOCATIONS*	FILE NO. 90B449C
	SCALE: AS SHOWN; DRAWN BY: J. RICKFORD DATE: 8/27/91; CHKD. BY: W Reed DATE: 10/25/91		FIG. NO. 4

APPENDIX A

APPENDIX A

PARTIAL LISTING OF TAXONOMIC REFERENCES FOR BENTHIC INVERTEBRATES

PARTIAL LISTING OF TAXONOMIC REFERENCES FOR BENTHIC INVERTEBRATES

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Bobb, M. L. 1974. The insects of Virginia no. 7: the aquatic and semi-aquatic Hemiptera of Virginia. Virginia Polytechnic Institute and State University, Blacksburg, Virginia. 195 p.

Bode, R. W. 1983. Larvae of North American Eukiefferiella and Tvetenia (Diptera: Chironomidae). New York State Mus. Bull. 452:1-40.

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APPENDIX B

PRELIMINARY SEDIMENT DATA
TARGET COMPOUND LIST (TCL) SEMIVOLATILE ORGANICS 00815

Compound	SG-C6 (µg/kg)	DSG-C6[2] (µg/kg)	SG-D6 (µg/kg)	SG-J6 (µg/kg)	SG-F7 (µg/kg)	SG-J7 (µg/kg)
DIBENZOFURAN	U	U	U	U	U	U
DIETHYLPHTHALATE	U	U	U	U	U	U
DIMETHYL PHTHALATE	U	U	U	U	U	U
FLUORANTHENE	91J	U	U	U	110J	U
FLUORENE	U	U	U	U	U	U
HEXACHLOROBENZENE	U	U	U	U	U	U
HEXACHLOROBUTADIENE	U	U	U	U	U	U
HEXACHLOROCYCLOPENTADIENE	U	U	U	U	U	U
HEXACHLOROETHANE	U	U	U	U	U	U
INDENO(1,2,3-CD)PYRENE	U	U	U	U	U	U
ISOPHORONE	U	U	U	U	U	U
N-NITROSO-DI-N-PROPYLAMINE	U	U	U	U	U	U
N-NITROSODIPHENYLAMINE (1)	U	U	U	U	U	U
NAPHTHALENE	U	U	U	U	U	U
NITROBENZENE	U	U	U	U	U	U
PENTACHLOROPHENOL	U	U	U	U	U	U
PHENANTHRENE	U	U	U	U	U	U
PHENOL	U	U	U	U	U	U
PYRENE	170J	U	U	U	230J	U

NOTES:

B Analyte was found in the associated blank as well as the sample.
C Compound confirmed by GC/MS.
D Result from a diluted sample.
E Concentrations exceeded the calibration range. The original extract was diluted and reanalyzed per the method.
J An estimated value below the Contract Required Quantitation Limit.
P Greater than 25 percent difference for detected concentrations between the two GC columns.
U Compound was analyzed for, but not detected.
X The detected concentrations from the two GC columns varied more than a factor of 2.
1 The designation DL indicates that the results are from reanalysis of the sample extract after dilution.
2 Field duplicate sample.

PRELIMINARY SEDIMENT DATA
TARGET COMPOUND LIST (TCL) SEMIVOLATILE ORGANICS 4 00816

Compound	SG-G8 (µg/kg)	SG-H8 (µg/kg)	SG-G9 (µg/kg)	SG-C10 (µg/kg)	SG-D10 (µg/kg)	SG-D10DL[1] (µg/kg)
1,2,4-TRICHLOROBENZENE	U	U	U	U	U	U
1,2-DICHLOROBENZENE	U	U	U	U	U	U
1,3-DICHLOROBENZENE	U	U	U	U	U	U
1,4-DICHLOROBENZENE	U	U	U	U	U	U
2,4,5-TRICHLOROPHENOL	U	U	U	U	U	U
2,4,6-TRICHLOROPHENOL	U	U	U	U	U	U
2,4-DICHLOROPHENOL	U	U	U	U	U	U
2,4-DIMETHYLPHENOL	U	U	U	U	U	U
2,4-DINITROPHENOL	U	U	U	U	U	U
2,4-DINITROTOLUENE	U	U	U	U	U	U
2,6-DINITROTOLUENE	U	U	U	U	U	U
2-CHLORONAPHTHALENE	U	U	U	U	U	U
2-CHLOROPHENOL	U	U	U	U	U	U
2-METHYLNAPHTHALENE	U	U	U	U	U	U
2-METHYLPHENOL	U	U	U	U	U	U
2-NITROANILINE	U	U	U	U	U	U
2-NITROPHENOL	U	U	U	U	U	U
3,3'-DICHLOROBENZIDINE	U	U	U	U	U	U
3-NITROANILINE	U	U	U	U	U	U
4,6-DINITRO-2-METHYLPHENOL	U	U	U	U	U	U
4-BROMOPHENYL-PHENYLETHER	U	U	U	U	U	U
4-CHLORO-3-METHYLPHENOL	U	U	U	U	U	U
4-CHLOROANILINE	U	U	U	U	U	U
4-CHLOROPHENYL-PHENYLETHER	U	U	U	U	U	U
4-METHYLPHENOL	U	U	U	U	U	U
4-NITROANILINE	U	U	U	U	U	U
4-NITROPHENOL	U	U	U	U	U	U
ACENAPHTHENE	U	U	U	U	U	U
ACENAPHTHYLENE	U	U	U	U	U	U
ANTHRACENE	U	U	U	U	U	U
BENZO(A)ANTHRACENE	U	U	U	U	U	U
BENZO(A)PYRENE	U	U	U	U	U	U
BENZO(B)FLUORANTHENE	U	U	U	U	U	U
BENZO(G,H,I)PERYLENE	U	U	U	U	U	U
BENZO(K)FLUORANTHENE	U	U	U	U	U	U
BIS(2-CHLOROETHOXY)METHANE	U	U	U	U	U	U
BIS(2-CHLOROETHYL)ETHER	U	U	U	U	U	U
BIS(2-CHLOROISOPROPYL)ETHER	U	U	U	U	U	U
BIS(2-ETHYLHEXYL)PHTHALATE	410J	U	270J	430J	34000E	37000DE
BUTYLBENZYLPHTHALATE	U	U	U	U	U	U
CARBAZOLE	U	U	U	U	U	U
CHRYSENE	U	U	U	U	U	U
DI-N-BUTYLPHTHALATE	U	U	U	U	U	U
DI-N-OCTYL PHTHALATE	U	U	U	U	U	U
DIBENZO(A,H)ANTHRACENE	U	U	U	U	U	U

PRELIMINARY SEDIMENT DATA
TARGET COMPOUND LIST (TCL) SEMIVOLATILE ORGANICS 3 4 00817

Compound	SG-G8 (µg/kg)	SG-H8 (µg/kg)	SG-G9 (µg/kg)	SG-C10 (µg/kg)	SG-D10 (µg/kg)	SG-D10DL[1] (µg/kg)
DIBENZOFURAN	U	U	U	U	U	U
DIETHYLPHTHALATE	U	U	U	U	U	U
DIMETHYL PHTHALATE	U	U	U	U	U	U
FLUORANTHENE	U	100J	U	U	U	U
FLUORENE	U	U	U	U	U	U
HEXACHLOROBENZENE	U	U	U	U	U	U
HEXACHLOROBUTADIENE	U	U	U	U	U	U
HEXACHLOROCYCLOPENTADIENE	U	U	U	U	U	U
HEXACHLOROETHANE	U	U	U	U	U	U
INDENO(1,2,3-CD)PYRENE	U	U	U	U	U	U
ISOPHORONE	U	U	U	U	U	U
N-NITROSO-DI-N-PROPYLAMINE	U	U	U	U	U	U
N-NITROSODIPHENYLAMINE (1)	U	U	U	U	U	U
NAPHTHALENE	U	U	U	U	U	U
NITROBENZENE	U	U	U	U	U	U
PENTACHLOROPHENOL	U	U	U	U	U	U
PHENANTHRENE	U	U	U	U	U	U
PHENOL	U	U	U	U	U	U
PYRENE	160J	250J	150J	180J	270J	U

NOTES:

B Analyte was found in the associated blank as well as the sample.
C Compound confirmed by GC/MS.
D Result from a diluted sample.
E Concentrations exceeded the calibration range. The original extract was diluted and reanalyzed per the method.
J An estimated value below the Contract Required Quantitation Limit.
P Greater than 25 percent difference for detected concentrations between the two GC columns.
U Compound was analyzed for, but not detected.
X The detected concentrations from the two GC columns varied more than a factor of 2.
1 The designation DL indicates that the results are from reanalysis of the sample extract after dilution.

PRELIMINARY SEDIMENT DATA
TARGET COMPOUND LIST (TCL) SEMIVOLATILE ORGANICS 3 4 00818

Compound	SG-I10 (µg/kg)	SG-BD05 (µg/kg)	SG-BD06 (µg/kg)
1,2,4-TRICHLOROBENZENE	U	U	U
1,2-DICHLOROBENZENE	U	U	U
1,3-DICHLOROBENZENE	U	U	U
1,4-DICHLOROBENZENE	290J	U	U
2,4,5-TRICHLOROPHENOL	U	U	U
2,4,6-TRICHLOROPHENOL	U	U	U
2,4-DICHLOROPHENOL	U	U	U
2,4-DIMETHYLPHENOL	U	U	U
2,4-DINITROPHENOL	U	U	U
2,4-DINITROTOLUENE	U	U	U
2,6-DINITROTOLUENE	U	U	U
2-CHLORONAPHTHALENE	U	U	U
2-CHLOROPHENOL	U	U	U
2-METHYLNAPHTHALENE	U	U	U
2-METHYLPHENOL	U	U	U
2-NITROANILINE	U	U	U
2-NITROPHENOL	U	U	U
3,3'-DICHLOROBENZIDINE	U	U	U
3-NITROANILINE	U	U	U
4,6-DINITRO-2-METHYLPHENOL	U	U	U
4-BROMOPHENYL-PHENYLETHER	U	U	U
4-CHLORO-3-METHYLPHENOL	U	U	U
4-CHLOROANILINE	U	U	U
4-CHLOROPHENYL-PHENYLETHER	U	U	U
4-METHYLPHENOL	U	U	U
4-NITROANILINE	U	U	U
4-NITROPHENOL	U	U	U
ACENAPHTHENE	U	U	U
ACENAPHTHYLENE	U	U	U
ANTHRACENE	U	U	U
BENZO(A)ANTHRACENE	U	U	U
BENZO(A)PYRENE	U	U	U
BENZO(B)FLUORANTHENE	U	U	U
BENZO(G,H,I)PERYLENE	U	U	U
BENZO(K)FLUORANTHENE	U	U	U
BIS(2-CHLOROETHOXY)METHANE	U	U	U
BIS(2-CHLOROETHYL)ETHER	U	U	U
BIS(2-CHLOROISOPROPYL)ETHER	U	U	U
BIS(2-ETHYLHEXYL)PHTHALATE	U	250BJ	240BJ
BUTYLBENZYLPHTHALATE	U	U	U
CARBAZOLE	U	U	U
CHRYSENE	U	U	U
DI-N-BUTYLPHTHALATE	U	U	U
DI-N-OCTYL PHTHALATE	U	U	U
DIBENZO(A,H)ANTHRACENE	U	U	U

3 4 00819

PRELIMINARY SEDIMENT DATA
TARGET COMPOUND LIST (TCL) SEMIVOLATILE ORGANICS

Compound	SG-I10 (µg/kg)	SG-BD05 (µg/kg)	SG-BD06 (µg/kg)
DIBENZOFURAN	U	U	U
DIETHYLPHTHALATE	U	U	U
DIMETHYL PHTHALATE	U	U	U
FLUORANTHENE	U	U	U
FLUORENE	U	U	U
HEXACHLOROBENZENE	1800	730J	4500E
HEXACHLOROBUTADIENE	U	U	U
HEXACHLOROCYCLOPENTADIENE	U	U	U
HEXACHLOROETHANE	U	U	U
INDENO(1,2,3-CD)PYRENE	U	U	U
ISOPHORONE	U	U	U
N-NITROSO-DI-N-PROPYLAMINE	U	U	U
N-NITROSODIPHENYLAMINE (1)	U	U	U
NAPHTHALENE	U	U	U
NITROBENZENE	U	U	U
PENTACHLOROPHENOL	U	U	U
PHENANTHRENE	U	U	U
PHENOL	U	U	U
PYRENE	U	U	U

NOTES:

B Analyte was found in the associated blank as well as the sample.
C Compound confirmed by GC/MS.
D Result from a diluted sample.
E Concentrations exceeded the calibration range. The original extract was diluted and reanalyzed per the method.
J An estimated value below the Contract Required Quantitation Limit.
P Greater than 25 percent difference for detected concentrations between the two GC columns.
U Compound was analyzed for, but not detected.
X The detected concentrations from the two GC columns varied more than a factor of 2.
1 The designation DL indicates that the results are from reanalysis of the sample extract after dilution.

3 4 00820

PRELIMINARY SEDIMENT DATA
TARGET COMPOUND LIST (TCL) PESTICIDE/PCB ANALYSIS

Compound	SG-C5 (µg/kg)	SG-C5DL[1] (µg/kg)	SG-C6 (µg/kg)	SG-C6DL[1] (µg/kg)	DSG-C6[2] (µg/kg)	DSG-C6DL[1,2] (µg/kg)
4,4'-DDD	110PXC	120PC	830PEC	760C	910PEC	890C
4,4'-DDE	120PC	100C	580PEC	530C	590PEC	590C
4,4'-DDT	U	52	170PE	120J	160P	140J
ALDRIN	19PX	28	U	U	U	U
ALPHA CHLORDANE	U	U	U	U	U	U
ALPHA-BHC	U	U	U	U	U	U
AROCLOR - 1016	U	U	U	U	U	U
AROCLOR - 1221	U	U	U	U	U	U
AROCLOR - 1232	U	U	U	U	U	U
AROCLOR - 1242	U	U	U	U	U	U
AROCLOR - 1248	U	U	U	U	U	U
AROCLOR - 1254	U	U	U	U	U	U
AROCLOR - 1260	U	U	U	U	U	U
BETA-BHC	U	U	15P	U	16PX	U
DELTA-BHC	U	U	U	U	54PX	U
DIELDRIN	U	U	U	U	U	U
ENDOSULFAN I	100P	110	60PEX	U	70PX	U
ENDOSULFAN II	20	U	U	U	U	U
ENDOSULFAN SULFATE	16P	U	U	U	U	U
ENDRIN	16PX	U	U	U	U	U
ENDRIN ALDEHYDE	18P	U	U	U	U	U
ENDRIN KETONE	U	U	U	U	U	U
GAMMA CHLORDANE	U	U	U	U	U	U
GAMMA-BHC	29	U	U	U	U	U
HEPTACHLOR	U	U	U	U	U	U
HEPTACHLOR EPOXIDE	3.0PX	U	U	U	U	U
METHOXYCHLOR	110P	U	U	U	U	U
TOXAPHENE	U	U	U	U	U	U

NOTES:

B Analyte was found in the associated blank as well as the sample.
C Compound confirmed by GC/MS.
D Result from a diluted sample.
E Concentrations exceeded the calibration range. The original extract was diluted and reanalyzed per the method.
J An estimated value below the Contract Required Quantitation Limit.
P Greater than 25 percent difference for detected concentrations between the two GC columns.
U Compound was analyzed for, but not detected.
X The detected concentrations from the two GC columns varied more than a factor of 2.
1 The designation DL indicates that the results are from reanalysis of the sample extract after dilution.
2 Field duplicate sample.

PRELIMINARY SEDIMENT DATA
TARGET COMPOUND LIST (TCL) PESTICIDE/PCB ANALYSIS 3 4 00821

Compound	SG-D6 (µg/kg)	SG-D6DL[1] (µg/kg)	SG-J6 (µg/kg)	SG-F7 (µg/kg)	SG-F7DL[1] (µg/kg)
4,4'-DDD	1900C	1300C	200C	440PEC	250C
4,4'-DDE	1200PC	850C	210PC	550PEC	380C
4,4'-DDT	550PE	290	84P	150P	79
ALDRIN	U	U	U	U	U
ALPHA CHLORDANE	U	U	U	U	U
ALPHA-BHC	24	U	U	U	U
AROCLOR - 1016	U	U	U	U	U
AROCLOR - 1221	U	U	U	U	U
AROCLOR - 1232	U	U	U	U	U
AROCLOR - 1242	U	U	U	U	U
AROCLOR - 1248	U	U	U	U	U
AROCLOR - 1254	U	U	U	U	U
AROCLOR - 1260	U	U	U	U	U
BETA-BHC	27PX	U	U	18P	U
DELTA-BHC	210PE	170P	U	U	87PX
DIELDRIN	36PX	U	15P	15P	U
ENDOSULFAN I	140PEX	U	17PX	51PEX	42PX
ENDOSULFAN II	U	U	U	U	U
ENDOSULFAN SULFATE	U	U	U	U	U
ENDRIN	U	U	U	U	U
ENDRIN ALDEHYDE	U	U	U	U	U
ENDRIN KETONE	U	U	U	U	U
GAMMA CHLORDANE	U	U	U	82X	U
GAMMA-BHC	U	U	U	U	U
HEPTACHLOR	U	U	U	U	U
HEPTACHLOR EPOXIDE	U	U	U	U	U
METHOXYCHLOR	U	U	U	U	U
TOXAPHENE	U	U	U	U	U

NOTES:

B Analyte was found in the associated blank as well as the sample.
C Compound confirmed by GC/MS.
D Result from a diluted sample.
E Concentrations exceeded the calibration range. The original extract was diluted and reanalyzed per the method.
J An estimated value below the Contract Required Quantitation Limit.
P Greater than 25 percent difference for detected concentrations between the two GC columns.
U Compound was analyzed for, but not detected.
X The detected concentrations from the two GC columns varied more than a factor of 2.
1 The designation DL indicates that the results are from reanalysis of the sample extract after dilution.

PRELIMINARY SEDIMENT DATA
TARGET COMPOUND LIST (TCL) PESTICIDE/PCB ANALYSIS 3 4 00822

Compound	SG-J7 (µg/kg)	SG-J7DL[1] (µg/kg)	SG-G8 (µg/kg)	SG-G8DL[1] (µg/kg)	SG-H8 (µg/kg)	SG-H8DL[1] (µg/kg)
4,4'-DDD	600PEC	510C	590PEC	570C	670PEC	580C
4,4'-DDE	750C	830PC	540PEC	530C	780EC	560PC
4,4'-DDT	320PE	310	200E	170	290PE	220
ALDRIN	U	U	U	U	U	U
ALPHA CHLORDANE	U	U	U	U	U	U
ALPHA-BHC	8.9P	U	U	U	U	U
AROCLOR - 1016	U	U	U	U	U	U
AROCLOR - 1221	U	U	U	U	U	U
AROCLOR - 1232	U	U	U	U	U	U
AROCLOR - 1242	U	U	U	U	U	U
AROCLOR - 1248	U	U	U	U	U	U
AROCLOR - 1254	U	U	U	U	U	U
AROCLOR - 1260	U	U	U	U	U	U
BETA-BHC	U	U	11P	U	U	U
DELTA-BHC	160PEX	130PX	54PX	54JP	U	U
DIELDRIN	7.9X	U	U	U	U	U
ENDOSULFAN I	69X	89PXE	45PEX	U	54PEX	U
ENDOSULFAN II	U	U	U	U	U	U
ENDOSULFAN SULFATE	U	U	U	U	U	U
ENDRIN	U	U	U	U	U	U
ENDRIN ALDEHYDE	U	U	U	U	U	U
ENDRIN KETONE	U	U	U	U	U	U
GAMMA CHLORDANE	U	U	U	U	U	U
GAMMA-BHC	U	U	U	U	U	U
HEPTACHLOR	U	U	U	U	U	U
HEPTACHLOR EPOXIDE	U	U	U	U	9.2PX	U
METHOXYCHLOR	U	U	U	U	U	U
TOXAPHENE	U	U	U	U	U	U

NOTES:

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X The detected concentrations from the two GC columns varied more than a factor of 2.
1 The designation DL indicates that the results are from reanalysis of the sample extract after dilution.

PRELIMINARY SEDIMENT DATA
TARGET COMPOUND LIST (TCL) PESTICIDE/PCB ANALYSIS

Compound	SG-G9 (µg/kg)	SG-G9DL[1] (µg/kg)	SG-C10 (µg/kg)	SG-C10DL[1] (µg/kg)	SG-D10 (µg/kg)	SG-D10DL[1] (µg/kg)
4,4'-DDD	430PEC	450C	920PEC	820C	990PEC	710C
4,4'-DDE	400PEC	410C	840PEC	760C	720PEC	520C
4,4'-DDT	180	190	150P	110J	580PE	360
ALDRIN	U	U	U	U	U	U
ALPHA CHLORDANE	U	U	U	U	U	U
ALPHA-BHC	U	U	U	U	U	U
AROCLOR - 1016	U	U	U	U	U	U
AROCLOR - 1221	U	U	U	U	U	U
AROCLOR - 1232	U	U	U	U	U	U
AROCLOR - 1242	U	U	U	U	U	U
AROCLOR - 1248	U	U	U	U	U	U
AROCLOR - 1254	U	U	U	U	U	U
AROCLOR - 1260	U	U	U	U	U	U
BETA-BHC	U	U	11PX	U	U	U
DELTA-BHC	33PX	U	52PX	U	30PX	U
DIELDRIN	U	U	U	U	U	U
ENDOSULFAN I	29X	U	92PX	U	64PEX	U
ENDOSULFAN II	U	U	U	U	U	U
ENDOSULFAN SULFATE	U	U	U	U	U	U
ENDRIN	U	U	U	U	U	U
ENDRIN ALDEHYDE	U	U	U	U	U	U
ENDRIN KETONE	U	U	U	U	U	U
GAMMA CHLORDANE	U	U	U	U	U	U
GAMMA-BHC	U	U	U	U	U	U
HEPTACHLOR	U	U	U	U	U	U
HEPTACHLOR EPOXIDE	U	U	U	U	U	U
METHOXYCHLOR	U	U	U	U	U	U
TOXAPHENE	U	U	U	U	U	U

NOTES:

B Analyte was found in the associated blank as well as the sample.
C Compound confirmed by GC/MS.
D Result from a diluted sample.
E Concentrations exceeded the calibration range. The original extract was diluted and reanalyzed per the method.
J An estimated value below the Contract Required Quantitation Limit.
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U Compound was analyzed for, but not detected.
X The detected concentrations from the two GC columns varied more than a factor of 2.
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PRELIMINARY SEDIMENT DATA
TARGET COMPOUND LIST (TCL) PESTICIDE/PCB ANALYSIS

Compound	SG-I10 (µg/kg)	SG-I10DL[1] (µg/kg)	SG-BD05 (µg/kg)	SG-BD05DL[2] (µg/kg)	SG-BD06 (µg/kg)	SG-BD06DL[1] (µg/kg)
4,4'-DDD	630PEC	560C	280PEC	300C	120PEC	110C
4,4'-DDE	1200EC	1400C	230PEC	260C	160EC	150C
4,4'-DDT	200	150	160PE	170	45	U
ALDRIN	U	U	U	U	U	U
ALPHA CHLORDANE	U	U	U	U	U	U
ALPHA-BHC	U	U	U	U	U	U
AROCLOR - 1016	U	U	U	U	U	U
AROCLOR - 1221	U	U	U	U	U	U
AROCLOR - 1232	U	U	U	U	U	U
AROCLOR - 1242	U	U	U	U	U	U
AROCLOR - 1248	U	U	U	U	U	U
AROCLOR - 1254	U	U	U	U	U	U
AROCLOR - 1260	U	U	U	U	U	U
BETA-BHC	8.8PX	U	U	U	U	U
DELTA-BHC	U	U	22PX	U	24PX	29PX
DIELDRIN	U	U	U	U	U	U
ENDOSULFAN I	150PEX	89PEX	U	U	U	U
ENDOSULFAN II	52	U	U	U	U	U
ENDOSULFAN SULFATE	U	U	U	U	U	U
ENDRIN	15PX	U	U	U	U	U
ENDRIN ALDEHYDE	U	U	U	U	U	U
ENDRIN KETONE	U	U	U	U	U	U
GAMMA CHLORDANE	U	U	U	U	U	U
GAMMA-BHC	U	U	U	U	U	U
HEPTACHLOR	U	U	U	U	U	U
HEPTACHLOR EPOXIDE	17P	U	U	U	U	U
METHOXYCHLOR	U	U	U	U	U	U
TOXAPHENE	U	U	U	U	U	U

NOTES:

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C Compound confirmed by GC/MS.
D Result from a diluted sample.
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PRELIMINARY SEDIMENT DATA
INORGANIC CONSTITUENTS

Compound	SG-G3 (mg/kg)	SG-H4 (mg/kg)	SG-K4 (mg/kg)	SG-C5 (mg/kg)	SG-C6 (mg/kg)	DSG-C6 (mg/kg)
ANTIMONY	4.8	9.1	2.6	3.7	6.9	2.2
ARSENIC	3.4	6.6	3.7	2.1	6.7	6.0
BERYLLIUM	1.0	1.3	0.8	0.9	1.9	2.2
CADMIUM	0.4	0.5	0.1	0.3	0.4	ND
CHROMIUM	21.3	44.7	30.8	10.5	26.9	29.6
COPPER	25.2	31.8	18.4	3.33	21.5	21.7
CYANIDE	ND[1]	0.47	ND	ND	ND	0.30
LEAD	16.5	28.5	19.5	5.9	26.7	22.4
MERCURY	12.9	21.6	2.8	5.3	7.0	6.9
NICKEL	11.5	20.9	14.1	2.67	13.5	13.9
SELENIUM	ND	ND	0.2	0.1	0.1	0.5
SILVER	0.87	1.18	0.41	0.13	0.38	0.87
THALLIUM	ND	ND	ND	ND	ND	ND
ZINC	89.7	13.3	83.0	10.3	104	112

NOTES:

1 ND = Not detected.

PRELIMINARY SEDIMENT DATA
INORGANIC CONSTITUENTS

Compound	SG-D6 (mg/kg)	SG-J6 (mg/kg)	SG-F7 (mg/kg)	SG-J7 (mg/kg)	SG-G8 (mg/kg)	SG-H8 (mg/kg)
ANTIMONY	3.0	6.5	24.6	6.3	6.0	9.5
ARSENIC	7.3	10.1	7.4	7.6	6.9	8.2
BERYLLIUM	2.5	2.5	2.5	1.2	2.0	2.3
CADMIUM	1.0	1.0	0.4	0.5	0.4	0.9
CHROMIUM	33.5	54.5	37.1	18.8	40.8	42.3
COPPER	20.5	45.5	35.0	21.4	28.4	35.9
CYANIDE	0.3	ND	ND	ND	ND	ND
LEAD	22.4	44.2	30.7	15.2	23.8	37.7
MERCURY	4.5	16.5	8.2	15.3	7.6	8.6
NICKEL	22.0	24.5	17.9	11.6	19.6	21.8
SELENIUM	2.4	0.7	0.5	0.6	0.4	0.8
SILVER	1.0	ND	0.42	0.23	0.8	0.9
THALLIUM	0.9	0.8	ND	ND	ND	ND
ZINC	123	205	170	89.1	152	202

PRELIMINARY SEDIMENT DATA
INORGANIC CONSTITUENTS

Compound	SG-G9 (mg/kg)	SG-C10 (mg/kg)	SG-D10 (mg/kg)	SG-I10 (mg/kg)	SG-BD05 (mg/kg)	SG-BD06 (mg/kg)
ANTIMONY	22.5	22.7	20.0	6.2	7.4	10.2
ARSENIC	7.1	8.3	8.4	12.8	4.1	1.6
BERYLLIUM	1.2	1.4	1.6	2.1	2.1	2.3
CADMIUM	0.9	0.1	0.7	0.4	0.5	0.1
CHROMIUM	29.54	35.9	43.2	45.4	39.2	18.5
COPPER	30.0	24.5	27.7	50.4	17.4	12.7
CYANIDE	ND	ND	0.28	ND	0.26	ND
LEAD	24.9	24.7	24.3	32.6	19.3	5.7
MERCURY	7.5	4.2	6.8	30.1	1.3	1.8
NICKEL	17.9	21.4	21.8	22.9	25.3	20.2
SELENIUM	0.7	0.8	0.7	1.2	0.8	0.2
SILVER	0.83	1.36	0.45	0.42	0.29	0.73
THALLIUM	ND	0.5	0.3	ND	ND	ND
ZINC	144	144	152	199	116	88.9

APPENDIX B

PRELIMINARY SEDIMENT DATA

PRELIMINARY SEDIMENT DATA
TARGET COMPOUND LIST (TCL) VOLATILE ORGANICS

3 4 00829

Compound	SG-G3 (µg/kg)	SG-G3DL[1] (µg/kg)	SG-H4 (µg/kg)	SG-K4 (µg/kg)
1,1,1-TRICHLOROETHANE	U	U	U	U
1,1,2,2-TETRACHLOROETHANE	U	U	U	U
1,1,2-TRICHLOROETHANE	U	U	U	U
1,1-DICHLOROETHANE	U	U	U	U
1,1-DICHLOROETHENE	U	U	U	U
1,2-DICHLOROPROPANE	U	U	U	U
1,2-DICHOLOROETHANE	U	U	U	U
1,2-DICHOLOROETHENE(TOTAL)	U	U	U	U
2-BUTANONE	U	U	U	U
2-HEXANONE	U	U	U	U
4-METHYL-2-PENTANONE	U	U	U	U
ACETONE	69B	93BD	240B	180B
BENZENE	U	U	U	U
BROMODICHLOROMETHANE	U	U	U	U
BROMOFORM	U	U	U	U
BROMOMETHANE	U	U	U	U
CARBON DISULFIDE	6J	U	13J	3J
CARBON TETRACHLORIDE	U	U	U	U
CHLOROBENZENE	920E	1000D	100	9J
CHLOROETHANE	U	U	U	U
CHOLOROFORM	U	U	U	U
CHOLOROMETHANE	U	U	U	U
CIS-1,3-DICHLOROPROPENE	U	U	U	U
DIBROMOCHLOROMETHANE	U	U	U	U
ETHYLBENZENE	U	U	U	U
METHYLENE CHLORIDE	22B	420BD	31B	45B
STYRENE	U	U	U	U
TETRACHLOROETHENE	U	U	U	U
TOLUENE	U	U	U	U
TRANS-1,3-DICHLOROPROPENE	U	U	U	U
TRICHLOROETHENE	U	U	U	U
VINYL CHLORIDE	U	U	U	U
XYLENE (TOTAL)	U	U	U	U

NOTES:

B Analyte was found in the associated blank as well as the sample.
C Compound confirmed by GC/MS.
D Result from a diluted sample.
E Concentrations exceeded the calibration range. The original extract was diluted and reanalyzed per the method.
J An estimated value below the Contract Required Quantitation Limit.
P Greater than 25 percent difference for detected concentrations between the two GC columns.
U Compound was analyzed for, but not detected.
X The detected concentrations from the two GC columns varied more than a factor of 2.
1 The designation DL indicates that the results are from analysis of the sample extract after dilution.

PRELIMINARY SEDIMENT DATA
TARGET COMPOUND LIST (TCL) VOLATILE ORGANICS

3 4 00830

Compound	SG-C5 (µg/kg)	SG-C5DL[1] (µg/kg)	SG-C6 (µg/kg)	DSG-C6[2] (µg/kg)
1,1,1-TRICHLOROETHANE	U	U	U	U
1,1,2,2-TETRACHLOROETHANE	U	U	U	U
1,1,2-TRICHLOROETHANE	U	U	U	U
1,1-DICHLOROETHANE	U	U	U	U
1,1-DICHLOROETHENE	U	U	U	U
1,2-DICHLOROPROPANE	U	U	U	U
1,2-DICHOLOROETHANE	U	U	U	U
1,2-DICHOLOROETHENE(TOTAL)	U	U	U	U
2-BUTANONE	U	U	64	140
2-HEXANONE	U	U	U	U
4-METHYL-2-PENTANONE	U	U	U	U
ACETONE	59B	100BD	470B	670B
BENZENE	U	U	U	U
BROMODICHLOROMETHANE	U	U	U	U
BROMOFORM	U	U	U	U
BROMOMETHANE	U	U	U	U
CARBON DISULFIDE	U	U	30J	27J
CARBON TETRACHLORIDE	U	U	U	U
CHLOROBENZENE	480E	460D	380	400
CHLOROETHANE	U	U	U	U
CHOLOROFORM	U	U	U	U
CHOLOROMETHANE	U	U	U	U
CIS-1,3-DICHLOROPROPENE	U	U	U	U
DIBROMOCHLOROMETHANE	U	U	U	U
ETHYLBENZENE	U	U	U	U
METHYLENE CHLORIDE	17B	35BDJ	43B	39BJ
STYRENE	U	U	U	U
TETRACHLOROETHENE	U	U	U	U
TOLUENE	U	U	U	U
TRANS-1,3-DICHLOROPROPENE	U	U	U	U
TRICHLOROETHENE	U	U	U	U
VINYL CHLORIDE	U	U	U	U
XYLENE (TOTAL)	U	U	U	U

NOTES:

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- J An estimated value below the Contract Required Quantitation Limit.
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- U Compound was analyzed for, but not detected.
- X The detected concentrations from the two GC columns varied more than a factor of 2.
- 1 The designation DL indicates that the results are from analysis of the sample extract after dilution.
- 2 Field duplicate sample.

PRELIMINARY SEDIMENT DATA
TARGET COMPOUND LIST (TCL) VOLATILE ORGANICS

3 4 00831

Compound	SG-D6 (µg/kg)	SG-J6 (µg/kg)	SG-F7 (µg/kg)	SG-J7 (µg/kg)
1,1,1-TRICHLOROETHANE	U	U	U	U
1,1,2,2-TETRACHLOROETHANE	U	U	U	U
1,1,2-TRICHLOROETHANE	U	U	U	U
1,1-DICHLOROETHANE	U	U	U	U
1,1-DICHLOROETHENE	U	U	U	U
1,2-DICHLOROPROPANE	U	U	U	U
1,2-DICHOLOROETHANE	U	U	U	U
1,2-DICHOLOROETHENE(TOTAL)	U	U	U	U
2-BUTANONE	U	31J	72	62
2-HEXANONE	U	U	U	U
4-METHYL-2-PENTANONE	U	U	U	U
ACETONE	540B	380B	320B	250B
BENZENE	U	U	U	U
BROMODICHLOROMETHANE	U	U	U	U
BROMOFORM	U	U	U	U
BROMOMETHANE	U	U	U	U
CARBON DISULFIDE	11J	18J	64	5J
CARBON TETRACHLORIDE	U	U	U	U
CHLOROBENZENE	650	10J	70	12J
CHLOROETHANE	U	U	U	U
CHOLOROFORM	U	U	U	U
CHOLOROMETHANE	U	U	U	U
CIS-1,3-DICHLOROPROPENE	U	U	U	U
DIBROMOCHLOROMETHANE	U	U	U	U
ETHYLBENZENE	U	U	U	U
METHYLENE CHLORIDE	55B	150B	35BJ	28B
STYRENE	U	U	U	U
TETRACHLOROETHENE	U	U	U	U
TOLUENE	U	U	U	U
TRANS-1,3-DICHLOROPROPENE	U	U	U	U
TRICHLOROETHENE	U	U	U	U
VINYL CHLORIDE	U	U	U	U
XYLENE (TOTAL)	U	U	U	U

NOTES:

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X The detected concentrations from the two GC columns varied more than a factor of 2.
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PRELIMINARY SEDIMENT DATA
TARGET COMPOUND LIST (TCL) VOLATILE ORGANICS

3 4 00832

Compound	SG-G8 (µg/kg)	SG-H8 (µg/kg)	SG-G9 (µg/kg)	SG-C10 (µg/kg)
1,1,1-TRICHLOROETHANE	U	U	U	U
1,1,2,2-TETRACHLOROETHANE	U	U	U	U
1,1,2-TRICHLOROETHANE	U	U	U	U
1,1-DICHLOROETHANE	U	U	U	U
1,1-DICHLOROETHENE	U	U	U	U
1,2-DICHLOROPROPANE	U	U	U	U
1,2-DICHOLOROETHANE	U	U	U	U
1,2-DICHOLOROETHENE(TOTAL)	U	U	U	U
2-BUTANONE	50	U	45	200
2-HEXANONE	U	U	U	U
4-METHYL-2-PENTANONE	U	U	U	U
ACETONE	600B	1000BE	350B	890B
BENZENE	U	U	U	U
BROMODICHLOROMETHANE	U	U	U	U
BROMOFORM	U	U	U	U
BROMOMETHANE	U	U	U	U
CARBON DISULFIDE	20J	U	23J	28J
CARBON TETRACHLORIDE	U	U	U	U
CHLOROBENZENE	12J	14J	39J	48
CHLOROETHANE	U	U	U	U
CHOLOROFORM	U	U	U	U
CHOLOROMETHANE	U	U	U	U
CIS-1,3-DICHLOROPROPENE	U	U	U	U
DIBROMOCHLOROMETHANE	U	U	U	U
ETHYLBENZENE	U	U	U	U
METHYLENE CHLORIDE	45B	46B	41BJ	33BJ
STYRENE	U	U	U	U
TETRACHLOROETHENE	U	U	U	U
TOLUENE	U	U	U	U
TRANS-1,3-DICHLOROPROPENE	U	U	U	U
TRICHLOROETHENE	U	U	U	U
VINYL CHLORIDE	U	U	U	U
XYLENE (TOTAL)	U	U	U	U

NOTES:

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- C Compound confirmed by GC/MS.
- D Result from a diluted sample.
- E Concentrations exceeded the calibration range. The original extract was diluted and reanalyzed per the method.
- J An estimated value below the Contract Required Quantitation Limit.
- P Greater than 25 percent difference for detected concentrations between the two GC columns.
- U Compound was analyzed for, but not detected.
- X The detected concentrations from the two GC columns varied more than a factor of 2.
- 1 The designation DL indicates that the results are from reanalysis of the sample extract after dilution.

PRELIMINARY SEDIMENT DATA
TARGET COMPOUND LIST (TCL) VOLATILE ORGANICS

3 4 00833

Compound	SG-D10 (µg/kg)	SG-I10 (µg/kg)	SG-BD05 (µg/kg)	SG-BD06 (µg/kg)
1,1,1-TRICHLOROETHANE	U	U	U	U
1,1,2,2-TETRACHLOROETHANE	U	U	U	U
1,1,2-TRICHLOROETHANE	U	U	U	U
1,1-DICHLOROETHANE	U	U	U	U
1,1-DICHLOROETHENE	U	U	U	U
1,2-DICHLOROPROPANE	U	U	U	U
1,2-DICHOLOROETHANE	U	U	U	U
1,2-DICHOLOROETHENE(TOTAL)	U	U	U	U
2-BUTANONE	140	60	34	U
2-HEXANONE	U	U	U	U
4-METHYL-2-PENTANONE	U	U	U	U
ACETONE	600B	220B	110B	59B
BENZENE	U	U	U	U
BROMODICHLOROMETHANE	U	U	U	U
BROMOFORM	U	U	U	U
BROMOMETHANE	U	U	U	U
CARBON DISULFIDE	56	17J	U	6J
CARBON TETRACHLORIDE	U	U	U	U
CHLOROBENZENE	45J	21J	42	46
CHLOROETHANE	U	U	U	U
CHOLOROFORM	U	U	U	U
CHOLOROMETHANE	U	U	U	U
CIS-1,3-DICHLOROPROPENE	U	U	U	U
DIBROMOCHLOROMETHANE	U	U	U	U
ETHYLBENZENE	U	U	U	U
METHYLENE CHLORIDE	45BJ	50B	58B	47B
STYRENE	U	U	U	U
TETRACHLOROETHENE	U	U	U	U
TOLUENE	U	U	U	U
TRANS-1,3-DICHLOROPROPENE	U	U	U	U
TRICHLOROETHENE	U	U	U	U
VINYL CHLORIDE	U	U	U	U
XYLENE (TOTAL)	U	U	U	U

NOTES:

- B Analyte was found in the associated blank as well as the sample.
- C Compound confirmed by GC/MS.
- D Result from a diluted sample.
- E Concentrations exceeded the calibration range. The original extract was diluted and reanalyzed per the method.
- J An estimated value below the Contract Required Quantitation Limit.
- P Greater than 25 percent difference for detected concentrations between the two GC columns.
- U Compound was analyzed for, but not detected.
- X The detected concentrations from the two GC columns varied more than a factor of 2.
- 1 The designation DL indicates that the results are from reanalysis of the sample extract after dilution.

PRELIMINARY SEDIMENT DATA
TARGET COMPOUND LIST (TCL) SEMIVOLATILE ORGANICS

Compound	SG-G3 (µg/kg)	SG-G3DL[1] (µg/kg)	SG-H4 (µg/kg)	SG-K4 (µg/kg)	SG-C5 (µg/kg)	SG-C5DL[1] (µg/kg)
1,2,4-TRICHLOROBENZENE	U	U	U	U	U	U
1,2-DICHLOROBENZENE	U	U	U	U	U	U
1,3-DICHLOROBENZENE	180J	U	U	U	200J	U
1,4-DICHLOROBENZENE	510J	U	U	U	470	U
2,4,5-TRICHLOROPHENOL	U	U	U	U	U	U
2,4,6-TRICHLOROPHENOL	U	U	U	U	U	U
2,4-DICHLOROPHENOL	U	U	U	U	U	U
2,4-DIMETHYLPHENOL	U	U	U	U	U	U
2,4-DINITROPHENOL	U	U	U	U	U	U
2,4-DINITROTOLUENE	U	U	U	U	U	U
2,6-DINITROTOLUENE	U	U	U	U	U	U
2-CHLORONAPHTHALENE	U	U	U	U	U	U
2-CHLOROPHENOL	U	U	U	U	U	U
2-METHYLNAPHTHALENE	U	U	U	U	U	U
2-METHYLPHENOL	U	U	U	U	U	U
2-NITROANILINE	U	U	U	U	U	U
2-NITROPHENOL	U	U	U	U	U	U
3,3'-DICHLOROBENZIDINE	U	U	U	U	U	U
3-NITROANILINE	U	U	U	U	U	U
4,6-DINITRO-2-METHYLPHENOL	U	U	U	U	U	U
4-BROMOPHENYL-PHENYLETHER	U	U	U	U	U	U
4-CHLORO-3-METHYLPHENOL	U	U	U	U	U	U
4-CHLOROANILINE	U	U	U	U	U	U
4-CHLOROPHENYL-PHENYLETHER	U	U	U	U	U	U
4-METHYLPHENOL	U	U	U	U	U	U
4-NITROANILINE	U	U	U	U	U	U
4-NITROPHENOL	U	U	U	U	U	U
ACENAPHTHENE	U	U	U	U	U	U
ACENAPHTHYLENE	U	U	U	U	U	U
ANTHRACENE	U	U	U	U	U	U
BENZO(A)ANTHRACENE	U	U	U	U	U	U
BENZO(A)PYRENE	U	U	U	U	U	U
BENZO(B)FLUORANTHENE	U	U	U	U	U	U
BENZO(G,H,I)PERYLENE	U	U	U	U	U	U
BENZO(K)FLUORANTHENE	U	U	U	U	U	U
BIS(2-CHLOROETHOXY)METHANE	U	U	U	U	U	U
BIS(2-CHLOROETHYL)ETHER	U	U	U	U	U	U
BIS(2-CHLOROISOPROPYL)ETHER	U	U	U	U	U	U
BIS(2-ETHYLHEXYL)PHTHALATE	240J	U	140J	U	2700E	2600DJ
BUTYLBENZYLPHTHALATE	U	U	U	U	U	U
CARBAZOLE	U	U	U	U	U	U
CHRYSENE	U	U	U	U	U	U
DI-N-BUTYLPHTHALATE	U	U	U	U	U	U
DI-N-OCTYL PHTHALATE	U	U	U	U	U	U
DIBENZO(A,H)ANTHRACENE	U	U	U	U	U	U

PRELIMINARY SEDIMENT DATA
TARGET COMPOUND LIST (TCL) SEMIVOLATILE ORGANICS

3 4 00835

Compound	SG-G3 (µg/kg)	SG-G3DL[1] (µg/kg)	SG-H4 (µg/kg)	SG-K4 (µg/kg)	SG-C5 (µg/kg)	SG-C5DL[1] (µg/kg)
DIBENZOFURAN	U	U	U	U	U	U
DIETHYLPHTHALATE	U	U	U	U	U	U
DIMETHYL PHTHALATE	U	U	U	U	U	U
FLUORANTHENE	U	U	U	U	U	U
FLUORENE	U	U	U	U	U	U
HEXACHLOROBENZENE	29000E	40000DE	500J	U	16000E	20000DE
HEXACHLOROBUTADIENE	U	U	U	U	U	U
HEXACHLOROCYCLOPENTADIENE	U	U	U	U	U	U
HEXACHLOROETHANE	U	U	U	U	U	U
INDENO(1,2,3-CD)PYRENE	U	U	U	U	U	U
ISOPHORONE	U	U	U	U	U	U
N-NITROSO-DI-N-PROPYLAMINE	U	U	U	U	U	U
N-NITROSODIPHENYLAMINE (1)	U	U	U	U	U	U
NAPHTHALENE	U	U	U	U	U	U
NITROBENZENE	U	U	U	U	U	U
PENTACHLOROPHENOL	U	U	U	U	U	U
PHENANTHRENE	U	U	U	U	U	U
PHENOL	U	U	U	U	U	U
PYRENE	U	U	U	U	U	U

NOTES:

B Analyte was found in the associated blank as well as the sample.
C Compound confirmed by GC/MS.
D Result from a diluted sample.
E Concentrations exceeded the calibration range. The original extract was diluted and reanalyzed per the method.
J An estimated value below the Contract Required Quantitation Limit.
P Greater than 25 percent difference for detected concentrations between the two GC columns.
U Compound was analyzed for, but not detected.
X The detected concentrations from the two GC columns varied more than a factor of 2.
1 The designation DL indicates that the results are from analysis of the sample extract after dilution.

PRELIMINARY SEDIMENT DATA
TARGET COMPOUND LIST (TCL) SEMIVOLATILE ORGANICS

3 4 00836

Compound	SG-C6 (µg/kg)	DSG-C6[2] (µg/kg)	SG-D6 (µg/kg)	SG-J6 (µg/kg)	SG-F7 (µg/kg)	SG-J7 (µg/kg)
1,2,4-TRICHLOROBENZENE	U	U	U	U	U	U
1,2-DICHLOROBENZENE	U	U	U	U	U	U
1,3-DICHLOROBENZENE	U	U	U	U	U	U
1,4-DICHLOROBENZENE	U	U	U	U	U	U
2,4,5-TRICHLOROPHENOL	U	U	U	U	U	U
2,4,6-TRICHLOROPHENOL	U	U	U	U	U	U
2,4-DICHLOROPHENOL	U	U	U	U	U	U
2,4-DIMETHYLPHENOL	U	U	U	U	U	U
2,4-DINITROPHENOL	U	U	U	U	U	U
2,4-DINITROTOLUENE	U	U	U	U	U	U
2,6-DINITROTOLUENE	U	U	U	U	U	U
2-CHLORONAPHTHALENE	U	U	U	U	U	U
2-CHLOROPHENOL	U	U	U	U	U	U
2-METHYLNAPHTHALENE	U	U	U	U	U	U
2-METHYLPHENOL	U	U	U	U	U	U
2-NITROANILINE	U	U	U	U	U	U
2-NITROPHENOL	U	U	U	U	U	U
3,3'-DICHLOROBENZIDINE	U	U	U	U	U	U
3-NITROANILINE	U	U	U	U	U	U
4,6-DINITRO-2-METHYLPHENOL	U	U	U	U	U	U
4-BROMOPHENYL-PHENYLETHER	U	U	U	U	U	U
4-CHLORO-3-METHYLPHENOL	U	U	U	U	U	U
4-CHLOROANILINE	U	U	U	U	U	U
4-CHLOROPHENYL-PHENYLETHER	U	U	U	U	U	U
4-METHYLPHENOL	U	U	U	U	U	U
4-NITROANILINE	U	U	U	U	U	U
4-NITROPHENOL	U	U	U	U	U	U
ACENAPHTHENE	U	U	U	U	U	U
ACENAPHTHYLENE	U	U	U	U	U	U
ANTHRACENE	U	U	U	U	U	U
BENZO(A)ANTHRACENE	U	U	U	U	U	U
BENZO(A)PYRENE	U	U	U	U	U	U
BENZO(B)FLUORANTHENE	U	U	U	U	U	U
BENZO(G,H,I)PERYLENE	U	U	U	U	U	U
BENZO(K)FLUORANTHENE	U	U	U	U	U	U
BIS(2-CHLOROETHOXY)METHANE	U	U	U	U	U	U
BIS(2-CHLOROETHYL)ETHER	U	U	U	U	U	U
BIS(2-CHLOROISOPROPYL)ETHER	U	U	U	U	U	U
BIS(2-ETHYLHEXYL)PHTHALATE	240J	U	U	U	280J	U
BUTYLBENZYLPHTHALATE	U	U	U	U	U	U
CARBAZOLE	U	U	U	U	U	U
CHRYSENE	U	U	U	U	U	U
DI-N-BUTYLPHTHALATE	U	U	U	U	U	U
DI-N-OCTYL PHTHALATE	U	U	U	U	U	U
DIBENZO(A,H)ANTHRACENE	U	U	U	U	U	U

APPENDIX C

FISH TISSUE SAMPLE PREPARATION

TECHNICAL OPERATING PROCEDURE

DATE: 04/02/91
REPLACES: ORIGINAL

PROCEDURE TITLE: Preparation of Fish Tissue for Analytical Determinations in the Laboratory

AREA OF APPLICABILITY: Hazleton Wisconsin, Inc.
Environmental Chemistry

SCOPE:

This document outlines the preparation of Fish samples prior to laboratory analysis. These preparation steps are compatible with the analysis of volatile organics, semivolatile organics, Pesticides/PCBs and the determination of heavy metals. These procedures may also be used in the preparation of other biological organisms for the same parameters.

EQUIPMENT AND MATERIALS:

- Teflon-coated or porcelain spatula
- Pyrex glass tray, 8x12x2 inch.
- Knife, heavy blade (or meat cleaver)
- Mallet, plastic faces, 2 to 3 lbs.
- Hobart Stainless Steel meat grinder
- Blender, stainless steel blade (glass or stainless steel blender container only, No plastic containers)
- 40 ml glass vials with teflon septum caps
- Wide-mouth 8-16 oz glass jars with teflon lined caps
- Dry ice or liquid Nitrogen
- Methanol
- Hexane, Pesticide grade

PROCEDURE:

Note: Preparation steps must be done in a clean environment (i.e. not in an extraction lab) when the determination of volatile organics is to be determined. All efforts should be made to minimize sources of contamination.

1.0 All equipment to be used in the preparation of tissue samples should be soap and water washed and then sequentially rinsed with hot water, milli-Q water, methanol, and hexane. This procedure should be repeated between samples before re-using the equipment.

2.0 Fish samples may be prepared with the following specifications:

- o Whole Fish
- o Fillet of Fish
- o Skin On
- o Skin Off

The type of preparation usually takes into account, the size of the fish, the species being investigated and the class of the compound or compounds to be determined. Be sure to clarify the specific requirements of the project sponsor before proceeding in the preparation of the fish.

3.0 To prepare the fish sample for analytical pretreatment, unwrap and weigh each fish. (Small fish, such as minnows are usually collected as composites and will represent a single composite sample). A total weight of 250 grams is the perferred mass required when a complete screening of the fish is to be performed (Volatile organics, Semivolatile organics, Pesticides, Metals).

4.0 Chop larger fish into 2 to 3-inch cubes, using either a sharp knife and mallet or a butcher saw.

5.0 Grind the fish cubes in a large commercial meat grinder that has been precooled by grinding dry ice or by rinsing with liquid Nitrogen. (Note: When dealing with small quantities of tissue such as minnows, use a blender instead of the meat grinder, to minimze the loss of tissue in the grinding process itself).

6.0 Thouroughly mix the ground material, using a porcelain or teflon coated spatula. Regrind and mix material two additional times. Clean out any material remaining in the grinder; add this to the sample and mix well.

7.0 Weigh three 5.0 g portions of the sample into 40 ml glass vials recording the tare weight of the vial and the final weight of the vial plus sample. Cap the vial with a teflon septum screw cap and store in a freezer until ready for volatile analysis.

8.0 Transfer the remaining fish sample to a glass container and store in a freezer for later subsampling and analysis.

APPROVED BY: _____ DATE _____

David C. Hills
Manager
Environmental Chemistry

REVIEWED BY: _____ DATE _____

Deborah L. Keller
Manager
Quality Assurance Unit

APPENDIX D

MERCURY ANALYTICAL PROCEDURES

OLIN CORPORATION
PRODUCT QUALITY AND ENVIRONMENTAL CONTROL DEPARTMENT
CHARLESTON, TENNESSEE

DETERMINATION OF TOTAL MERCURY IN FISH

1.0 THEORY

1.1 This is a cold-vapor atomic absorption procedure at 253.7-nm. wavelength for the determination of total mercury in fish. After digestion, the mercury is reduced to the elemental state and aerated from solution in an open system. The mercury is passed through a cell positioned in the light path of an atomic absorption spectrometer. The absorbance (peak height) is measured as a function of mercury concentration.

Also, the method can be utilized for total mercury analyses of other biota.

2.0 SAFETY

2.1 Familiarize yourself with all equipment and chemicals used in this procedure according to the manufacturer's instructions.
Maintain an awareness of the danger associated with the various types of reagent chemicals used in this procedure.

2.2 All spills must be cleaned immediately and in the case of skin contact, wash with large volumes of cold water.

2.3 Because mercury vapor is toxic, precaution must be taken to avoid its inhalation. Therefore, the mercury vapor discharge from the cell must be vented into an exhaust hood or be passed through some type of absorbing medium.

2.4 Exhaust hood used for the digestion procedure must be approved for perchloric acid fumes.

3.0 APPARATUS

3.1 Atomic absorption spectrophotometer or equivalent: any atomic absorption spectrophotometer with an open sample presentation area in which to mount the absorption cell is suitable. Commercial instruments

are available using the cold-vapor technique and may be substituted for the atomic absorption spectrophotometer.

3.2 Exhaust hood: An approved perchloric acid hood.

3.3 Hot Plate: Variable thermostatic controller with a specific power of at least 1.7 watts/cm^2.

3.4 Flowmeter: Capable of measuring an air flow of one-liter/min.

3.5 Drying tube: 6-inch x 3/4-inches in diameter tube containing 10-20 grams of magnesium perchlorate.

3.6 Extension column: 7 1/4 inches long with 19/38 ground glass fitting.

3.7 Volumetric flask: 100 ml with 19/38 ground glass fitting.

3.8 BOD flask: 250 ml or equivalent with proper ground glass fitting.

3.9 Pipettes: Various sizes of glass pipettes

3.10 Recorder: Any multirange variable-speed recorder that is compatible with the atomic absorption UV detector system.

3.11 Cold-vapor Generator: See diagram 3.11

4.0 REAGENTS

4.1 Aqua Regia: Prepare daily three volumes of concentrated hydrochloric acid and one volume of concentrated nitric acid.

4.2 Nitric Acid, HNO_3: Concentrated, Reagent Grade

4.3 Perchloric Acid, $HClO_4$: 98 Percent, Reagent Grade

4.4 Hydrochloric Acid, HCL: Concentrated, Reagent Grade

4.5 D.I. Water, ASTM Type II

4.6 Potassium Dichromate, $K_2Cr_2O_7$: .02 Percent W/V, dissolve 0.2 grams of $K_2Cr_2O_7$ in D.I. Water and dilute to 1000 mL volume with D. I. water.

4.7 Stannous Chloride, $SnCl_2$: 10 Percent W/V; dissolve 119 grams of reagent grade $SnCl_2$ $2H_2O$ in 40 mL of concentrated hydrochloric acid and dilute to a liter volume with D.I. water.

4.8 Potassium Permanganate, $KmNO_4$: 5% W/V; dissolve 50 grams of $KmNO_4$ in 1,000 mL D. I. Water.

4.9 Magnesium perchlorate, $Mg(ClO_4)_2$; Anhydrous reagent grade crystals.

4.10 Mercury Standard, Hg: 1,000 µg/mL, dissolve 1.354 grams of $HgCl_2$ in 750 mL of D. I. water. Add 100 mL of concentrated nitric acid and dilute to a one-liter volume with D. I. Water. Commercial mercury standards are available and may be substituted for the above mercury standard.

4.11 Hydroxylamine hydrochloride, H_2NOH HCl: 4% W/V, dissolve 20 grams of reagent grade H_2NOH HCl in 60-70 mL D.I. Water. Adjust the pH of the solution to 2.8 + .2 with ammonium hydroxide, NH_4OH. Add 10 mL of a 4% sodium diethyldithiocarbamate solution to complex any metallic impurities. After five minutes, extract the metallic impurities with 20 mL of chloroform in a separatory funnel. Repeat the chloroform extraction twice discarding the chloroform layer after each extraction. Adjust the pH of the aqueous layer to 1.2 with hydrochloric acid and dilute to 500 mL volume with D.I. Water.

4.12 Absorbing Medium for mercury removal:

A. 0.25% (W/V) - iodine in a 3% (W/V) potassium iodide solution, or

B. equal volumes of 0.1 M $KmNO_4$ solution and 10% (W/V) H_2SO_4 Solution.

5.0 PROCEDURE

5.1 Standard Procedure

Prepare daily a 10µg/mL mercury solution by diluting a 10 mL aliquot of the 1,000 µg/mL mercury standard solution and 15 mL of concentrated nitric acid to a liter volume with D.I. Water.

Prepare a 0.1 µg/mL mercury solution by diluting a 1 mL aliquot of the 10µg/mL mercury solution and 15 mL of concentrated nitric acid to a volume of 100 mL with D.I. Water. Transfer 0, 0.5, 1, 3, and 5 mL aliquots of the 0.1 µg/mL mercury solution into a series of BOD flasks. Add 5 mL of freshly prepared aqua regia, 50 mL D.I. Water, 15 mL of $KmNO_4$ solution to each flask and heat thirty minutes in a water bath at 95 degrees Celsius. Allow the solutions to cool and add 6 mL of hydroxylamine hydrochloride solution to reduce the excess potassium permanganate to each flask. Add enough D.I. water to each flask to yield a total volume of 125 mL and analyze each aliquot by cold-vapor procedure.

5.2 Sample Preparation

Samples are individually filleted. Filet only is retained and kept frozen until ready for analysis. Allow filet to reach room temperature, place in a 250 ml beaker and homogenize thoroughly using Tissuemizer. Clean all associated equipment with 10 percent nitric acid to eliminate elevated cross-contamination after each sample is tissuemized.

5.3 Digestion Procedure

Preheat three hot plates in an approved perchloric acid exhaust hood to surface temperatures of 230, 265, and 340 ±5 degrees Celsius respectively.

Weigh approximately three grams of filet to the nearest .01 grams into a 100 mL volumetric flask. Add one mL of the $K_2Cr_2O_7$ Solution, 15 mL HNO_3, and 15 mL $HClO_4$ to each aliquot flask, mixing gently between each addition. Attach an extension column and place the apparatus on the first hot plate (See Diagram 5.2 attached) with a surface temperature of 230 degrees centigrade. Allow the apparatus to remain for 20-30 minutes or until all Nitrous Oxide fumes have visually ceased to evolve.

Place the apparatus on the second hot plate (surface temperature of 265 degrees Celsius) and allow it to remain there until a color change from yellow to green occurs.

Place the apparatus on the third hot plate (surface temperature at 340 degrees Celsius) and allow it to remain there until a color change from green to orange occurs.

Remove the apparatus from the hot plate and let cool to room temperature.

Remove the extension column and wash with D.I. water, collecting the washings in the 100 mL volumetric flask.

Bring to a volume of 100 mL with D.I. Water.

Pipette an aliquot containing an estimated .05 - .5 μg of mercury into a BOD flask. Add enough D.I. Water to make a total volume of 125 mL. The aliquot is now analyzed by cold-vapor atomic absorption spectrophotometer.

5.4 Auto-Digestion Procedure

The Charleston laboratory utilizes a Prolabo "Microdigest 300" microwave system which consists of an automated, computer controlled, 16-sample turntable capable of controlling various digestion parameters within very close tolerance.

Single laboratory analytical comparison studies between these two digestion procedures on forty-one samples with a range of 0.25 - 2.0 mg/kg showed a -0.025 mg/kg bias and 0.99 correlation coefficient. Also, a certified SRM (DORM-1) sample was analyzed which showed a 98.2 percent accuracy, -0.015 mg/kg bias and 6.4 percent RSD.

Weigh approximately one gram of filet to the nearest 0.01 grams into an auto-digestion flask, connect condenser, and place the flask into the sample turntable.

A duel treatment process (digital storage: SRM7B) utilizes a 3 ml HNO_3, 5 ml H_2SO_4, 5 percent power rate for seven minutes and a 1 ml $HClO_4$ and 5 percent power rate for three minutes.

Refer to the Operation Manual for complete operation of the Prolabo "Microdigest 300."

After digestion is completed, dilute to a 50 ml volume with DI water.

Pipette an aliquot containing an estimated .05 - .5 µg of mercury into a BOD flask. Add enough D.I. Water to make a total volume of 125 mL. The aliquot is now analyzed by cold-vapor atomic absorption spectrophotometer.

5.5 Cold-Vapor Procedure

Add 5 mL of stannous chloride solution; immediately attach the BOD bottle to the aeration apparatus. The sample is allowed to stand quietly without manual agitation. The nitrogen purge which has previously been adjusted to a rate of one liter/minute is allowed to purge the sample continuously. The absorbance will increase and reach a maximum within 30 seconds.
As soon as the recorder base line or levels pen levels off (approximately one minute), disconnect BOD bottle and continue to purge the cell with Nitrogen. Record the maximum peak height and calculate the concentration of mercury from the predetermined five-point calibration curve.

6.0 CALCULATIONS

6.1 Measure the peak height of the unknown from the chart and read the mercury value from the standard curve.

6.2 Calculate the mercury concentration in the sample by the formula:

$$\mu g\ Hg/gram = \frac{\mu g\ Hg\ in\ aliquot}{wt.\ of\ aliquot\ in\ gms}$$

6.3 Report mercury concentrations as follows:

Below 0.1 µg/gm, <0.1 µg; between 0.1 and 1 µg/gm, to nearest 0.01 µg; between 1 and 10 µg/gm, to nearest 0.1 µg; above 10 µg/gm, to nearest µg.

7.0 QUALITY ASSURANCE

7.1 All quality assurance data should be maintained, available for easy reference or inspection, and reported with the results.

7.2 A reagent blank must be analyzed with each batch or every twenty samples whichever is greater.

7.3 A sample duplicate must be analyzed with each batch or grouping of ten (10) samples or less.

7.4 A sample spike must be analyzed with each batch or with each grouping of ten (10) samples or less. Percent recovery for the spikes should be within the 70 - 130 percent range.

7.5 A check standard should be analyzed at the beginning of a batch of samples and after each set of ten sample runs. The percent delta or change in concentration should be less than 15 percent between each check standard.

Written by: H. B. Cochran

Approved by: J. P. Newman

4.HBC/cb

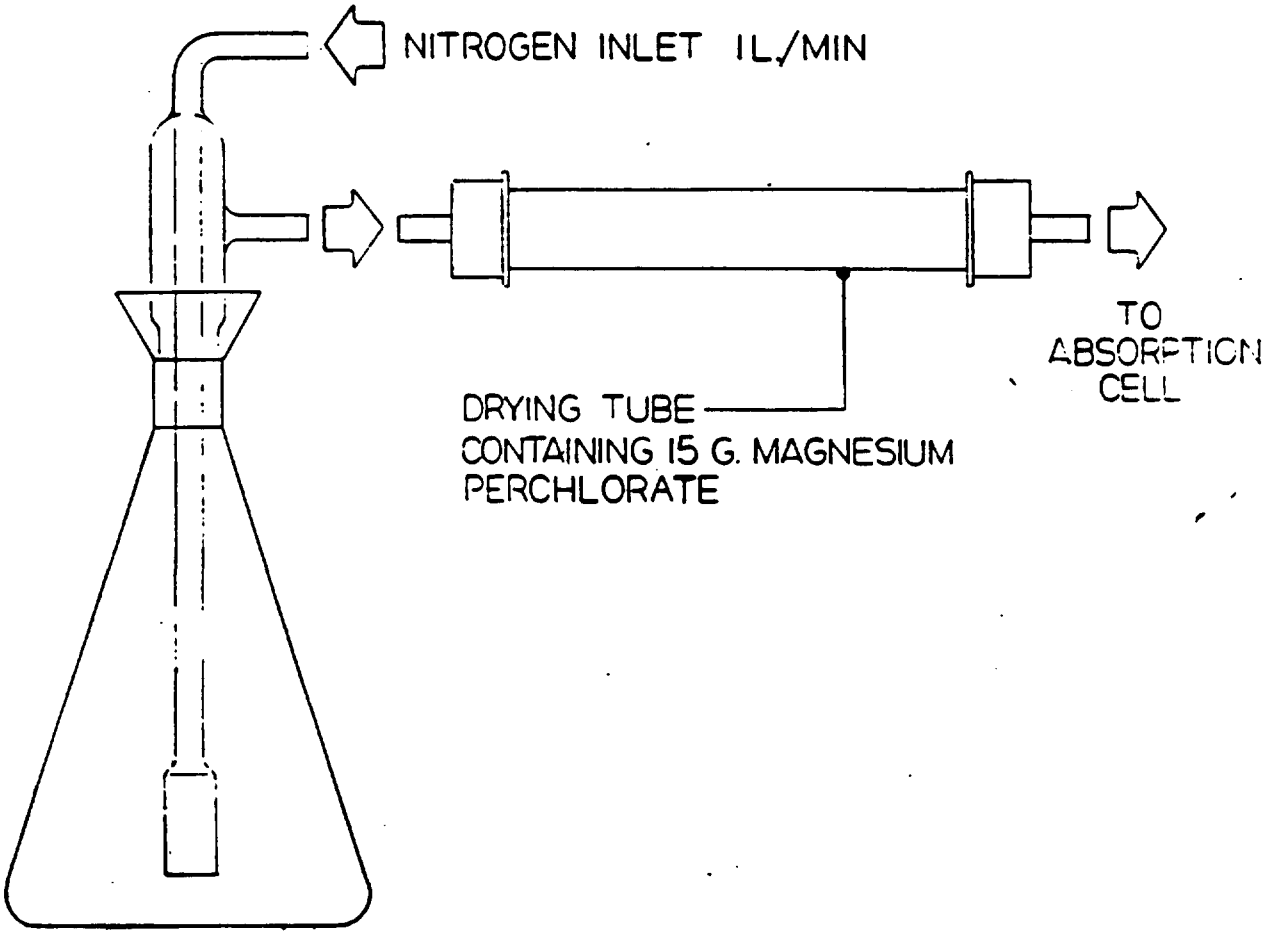
NITROGEN INLET 1L/MIN
TO ABSORPTION CELL
DRYING TUBE CONTAINING 15 G. MAGNESIUM PERCHLORATE

Diagram 3.11

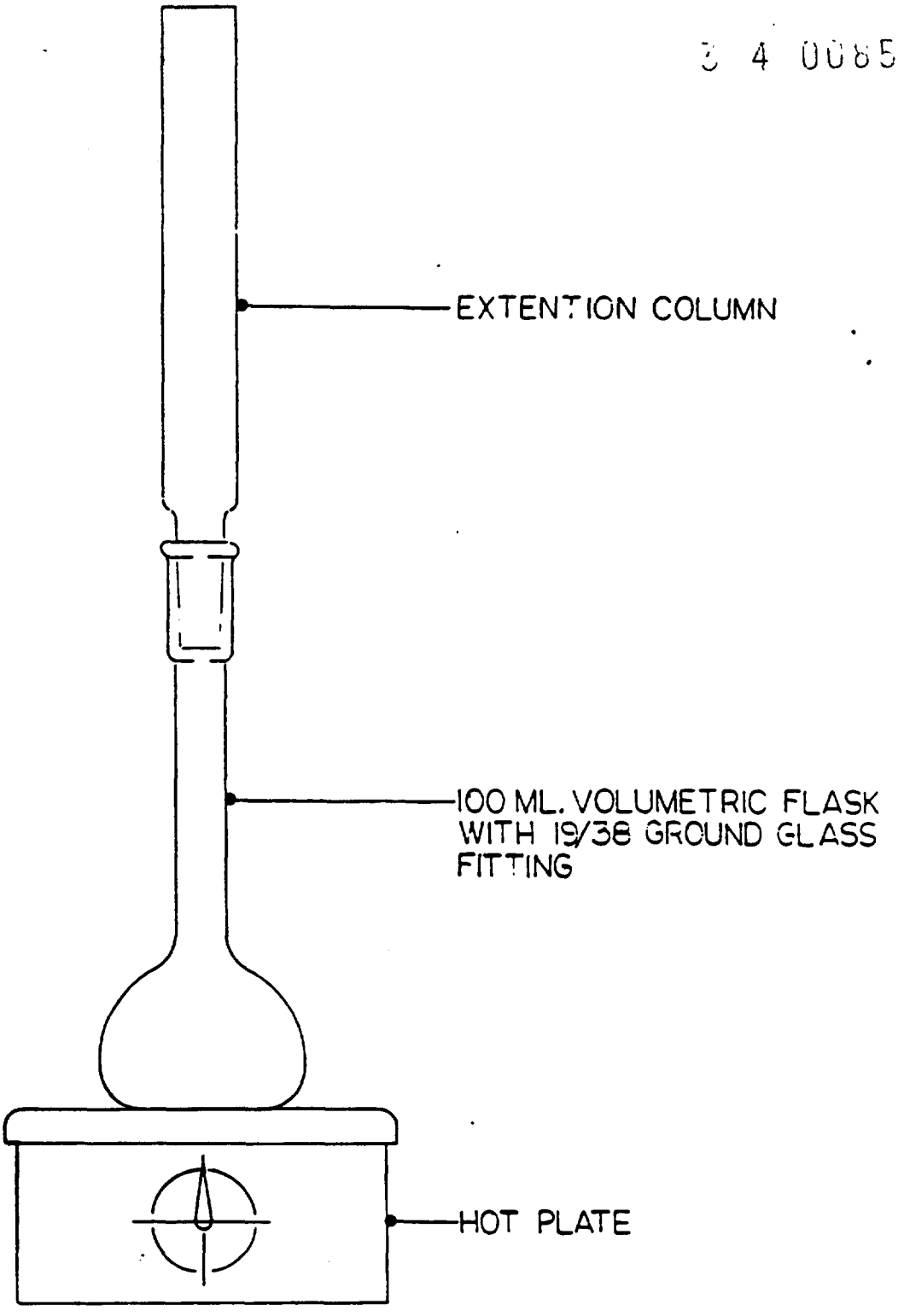
EXTENTION COLUMN
100 ML. VOLUMETRIC FLASK WITH 19/38 GROUND GLASS FITTING
HOT PLATE

Diagram 5.2

APPENDIX E

VOLATILE CHLORINATED BENZENE ANALYTICAL PROCEDURES

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Method Summary for Chlorobenzene in Fish Tissue by GC/MS

The analysis of chlorobenzene in fish tissue is performed in accordance with the US EPA 2/88 CLP protocol for soil analysis with modifications as follows:

Sample Preparation: All samples will be stored in a walk in freezer until sample preparation is performed. The individual fish samples will be homogenized in order to obtain a representative sample. The homogenizing will be performed with dry ice / liquid nitrogen to reduce volatilization that could occur during sample preparation.

Sample Analysis: Each sample will have 10uL of food grade anti-foam agent added to the sample immediately prior to analysis. This will not introduce any chlorobenzene to the analysis.

Only one internal standard, d5-Chlorobenzene, will be used for quantitation purposes. Only one surrogate standard will be utilized, bromofluorobenzene, with the recovery range equal to the recovery range of soil.

The samples will have matrix spike / matrix spike duplicate performed using only chlorobenzene as the spiking compound. The QC criteria will remain consistent with EPA CLP soil limits.

Detection Limit: The detection limit for chlorobenzene in fish is 5 ug/Kg.

REFERENCE:

1. Environmental Protection Agency Contract Laboratory Program Statement of Work for "Organic Analysis Multi-Media Multi-Concentration, " (February 1988). Exhibits B, D, E, Volatile Sections.

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APPENDIX F

SEMIVOLATILE CHLORINATED BENZENE, CHLORINATED PESTICIDES AND LIPIDS ANALYTICAL PROCEDURES

ASSAY TITLE: Semivolatile Analysis of Fish Samples by Gas Chromatography/Mass Spectroscopy (GC/MS)

AREA OF APPLICABILITY: Hazleton Wisconsin, Inc. Environmental Analysis

SCOPE:

This method is applicable to the determination of residues of the U.S. Environmental Protection Agency (EPA) - defined priority pollutants and target compound list (TCL) semivolatile organic compounds in fish and other aquatic biota. Analysis is conducted using GC-MS. Applicable analytes and their target detection limits are listed in Attachment 1.

PRINCIPLE:

Aliquots of homogenized sample are spiked with a surrogate standard mixture, dried by mixing with anhydrous sodium sulfate, and extracted with methylene chloride in soxhlet extractors. The methylene chloride extracts are purified by gel permeation chromatography (GPC), followed by florisil chromatography. The extracts are then concentrated, and an internal standard mixture is added as a quantification aid for subsequent analysis by GC-MS. Analytes are quantified versus calibration factors obtained from concurrently analyzed standards. GC-MS calibration is done according to guidelines given in the protocols of the EPA Contract Laboratory Program.

SENSITIVITY:

The sensitivity of this method depends on the level of interference within a given matrix.

Target quantification limits are listed in Attachment 1.

PRECISION AND ACCURACY:

The method is capable of providing precise and accurate determinations of analytes as summarized in Attachment 2 for spiked control fish. The data in Attachment 2 were acquired along with analysis of field samples between November 1987 and November 1988.

REFERENCES:

Federal Register, 49(209): 43385-43406, Environmental Protection Agency (EPA) Method 625 (October 16, 1984).

EPA Contract Laboratory Program, Statement of Work for "Organic Analysis Multi-Media Multi-Concentration" (October 1986). Revisions: January 1987; February 1987; July 1987; August 1987. Exhibits: B, D, E.

Methods 3540, 3640, and 8270, "Test Methods for Evaluating Solid Waste," EPA Publication SW-846, Third Edition, Washington, DC, Rev. 0 (September 1986).

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SAFETY PRECAUTIONS:

- The toxicity and carcinogenicity of the chemicals used in this method have not been precisely defined; each chemical should be treated as a potential health hazard and exposure to these chemicals should be minimized. Each analyst is responsible for maintaining awareness of OSHA regulations regarding safe handling of chemicals used in this method. Additional references to laboratory safety are available for the information of the analyst.

- The following parameters covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, and N-nitrosodimethylamine. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

- Observe all standard laboratory safety procedures as outlined in the Hazleton Wisconsin safety training manual.

INTERFERENCES:

Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample-processing hardware that lead to discrete artifacts or elevated baselines in the total ion current profiles (TICPs). All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. Matrix interferences may be caused by excess lipids in the sample extract or by contaminants that are extracted from the sample. The extent to which the matrix causes interferences will vary considerably from source to source. Cleanup procedures provided in this method may be used to minimize such interferences.

QUALITY ASSURANCE:

This section outlines the minimum quality control operations necessary to satisfy the analytical requirements associated with the determination of semivolatile organic TCL compounds in water.

1. Tuning and GC/MS mass calibration

   It is necessary to establish that a given GC/MS meets the standard mass spectral abundance criteria before initiating any ongoing data collection. This is accomplished through the analysis of decafluorotriphenylphosphine (DFTPP).

1.1 Each GC/MS system used for analysis of semivolatile compounds must be hardware-tuned to meet the abundance criteria listed in Attachment 3 for a 50-ng injection of DFTPP. DFTPP may be analyzed separately or as part of the calibration standard. The criteria must be demonstrated daily or for each 12-hour period, whichever is more frequent, before samples can be analyzed. DFTPP must be injected to meet this criterion.

1.2 Whenever corrective action is taken that may change or affect the tuning criteria for DFTPP (e.g., ion source cleaning or repair, etc.), the tuning must be verified regardless of the 12-hour tuning requirements.

2. Calibration of the GC/MS system

Before the analysis of samples and required blanks and after meeting the tuning criteria, the GC/MS system must be initially calibrated at a minimum of three concentrations to determine the linearity of response using TCL compound standards. Once the system has been calibrated, the calibration must be verified each 12-hour time period for each GC/MS system.

2.1 Prepare calibration standards containing all semivolatile target compounds (Attachment 10) at concentrations of 20, 50, and 100 µg/mL. Initial calibration is then performed by injecting 1 µL of each standard concentration, producing on-column injections of 20, 50, and 100 total ng.

2.2 Analyze each calibration standard and tabulate the area of the primary characteristic ion (Attachments 4 and 5) against concentration for each compound, including all required surrogate compounds. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time limits. Late-eluting compounds will usually have much better agreement.

2.2.1 Using Attachment 6, calculate the relative response factor (RRF) for each compound at each concentration level using the following Equation.

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured.

A_{is} = Area of the characteristic ion for the specific internal standards from Attachment 4 or 5.

C_{is} = Concentration of the internal standard (ng/µL).

C_x = Concentration of the compound to be measured (ng/µL).

2.2.2 Using the RRFs from the initial calibration, calculate the percent of relative standard deviation (%RSD) for compounds labeled as 2 calibration check compounds (CCCs) and shown in Attachment 6 using the following equation.

$$\% RSD = \frac{SD}{\bar{X}} \times 100$$

Where:

RSD = relative standard deviation

SD = standard deviation of initial response factors (per compound)

Where: $$SD = \sqrt{\sum_{i=1}^{N} \frac{(x_i - \bar{X})^2}{N-1}}$$

$\bar{X}$ = Mean of initial relative response factors (per compound)

2.2.3 The %RSD for each CCC (Attachment 7) must be <u>less</u> than or equal to 30.0%. These criteria must be met for the initial calibration to be valid.

2.3 A system performance check must be done to ensure that minimum average RRF are met before the calibration curve is used.

2.3.1 For semivolatiles, the system performance check compounds (SPCCs) are N-Nitroso-Di-n-Propylamine, Hexachlorocyclopentadiene, 2,4-Dinitrophenol, and 4-Nitrophenol. The minimum acceptable average RRF for these compounds is 0.050. SPCCs typically have very low RRFs (0.1 to 0.2) and tend to decrease in response as the chromatographic system or the standard material begins to deteriorate. These compounds are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated.

2.3.2 The initial calibration is valid only after both the %RSD for CCCs and the minimum RRF for SPCCs have been met. Only after both these criteria are met can sample analysis begin.

2.4 Continuing calibration

Continuing calibration is performed by injecting 1 µL of a 50 µg/mL standard containing all semivolatile TCL compounds, including all required surrogates, and is performed every 12 hours when samples are analyzed. Compare the RRF data from the standard every 12 hours with the average RRF from the initial calibration for a specific instrument. A system performance check must be made every 12 hours. If the SPCC criteria are met, a comparison of relative response factors is made for all compounds. This is the same check that is applied during the initial calibration. If the minimum RRFs are not met, the system must be evaluated and corrective action must be taken before sample analysis begins.

2.4.1 Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatography system. This check must be met before analysis begins. The minimum RRF for semivolatile SPCC is 0.050.

2.4.2 Calibration check compounds (CCCs)

After the system performance check is met, the CCCs listed in Attachment 7 are used to check the validity of the initial calibration. Calculate the percent difference using the following Equation.

$$\% \text{ Difference} = \frac{RRF_i - RRF_c}{RRF_i}$$

Where:

RRF_i = Average response factor from initial calibration

RRF_c = Response factor from current verification check standard.

2.4.3 If the percent difference for any compound is greater than 20%, this should be considered a warning limit. If the percent difference for each CCC is less than or equal to 30%, the initial calibration is assumed to be valid. If the criteria are not met (>30% difference) for any CCC, corrective action must be taken.

2.5 Documentation

Calculate and report the RRF and percent difference (%D) for each compound. Ensure that the minimum RRF for semivolatile SPCCs is 0.050. The %D for each CCC compound must be less than or equal to 30.0%.

3. Internal standard evaluation

Internal standard responses and retention times in all samples must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12-hour) calibration standard, the chromatographic system must be inspected for malfunctions and corrections must be made as required. The extraction ion current profile (EICP) area of the internal standards must be monitored and evaluated for each sample, blank, matrix spike, and matrix spike duplicate. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction and corrections must be made as appropriate. When corrections are made, the samples that were analyzed while the system was malfunctioning must be reanalyzed.

4. Method blank analysis

A method blank is a 30 g sample of sodium sulfate that is carried through the entire analytical scheme (extraction, concentration, and analysis). This information should be well documented and kept on file.

A method blank is analyzed with every 20 samples processed or whenever samples are extracted, whichever is most frequent. It is the analyst's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, and glassware are minimized. An acceptable laboratory blank should contain less than or equal to the reported method detection limit of any single target compound, or in the case of the phthalate esters, less than or equal to five times (5x) the respective detection limit.

If a laboratory method blank exceeds the above criteria, the analyst must consider the analytical system to be out of control. The source of the contamination must be investigated, and appropriate corrective action must be taken.

5. Surrogate spike (SS) analysis

Surrogate standard recoveries are determined for all samples and blanks. All samples and blanks are fortified with surrogate spiking compounds before extraction in order to monitor sample preparation and analysis.

5.1 Each sample, matrix spike, matrix spike duplicate, and blank are spiked with surrogate compounds before extraction. The surrogate spiking compounds shown in Attachment 8 are used to fortify each sample, matrix spike, matrix spike duplicate, and blank with the proper concentrations.

5.2 Surrogate spike recovery must be evaluated by determining whether the concentration (measured as percent recovery) falls inside the recovery limits listed in Attachment 9.

5.3 If the recovery of any surrogate compound in <u>either</u> base neutral or acid fraction is below 10%, or if the recoveries of two surrogate compounds in <u>either</u> base neutral or acid fractions are outside surrogate spike recovery limits, the analyst will document (i.e., record and discuss the problem and corrective action taken in the case narrative) and deviations from acceptable quality control limits and take the following actions:

5.3.1 Check calculations to ensure that there are no errors; check internal standard and surrogate spiking solutions for degradation, contamination, etc.; and check instrument performance.

5.3.2 If Step 5.3.1 fails to reveal a problem, then reanalyze the extract. If reanalysis of the extract solves the problem, then the problem was within the analyst's control. Therefore, only submit data from the analysis with surrogate spike recoveries within the control windows. This will be considered the initial analysis and will be reported as such on all data deliverables.

5.3.3 If Step 5.3.2 fails to solve the problem, then reextract and reanalyze the sample. If the reextraction and reanalysis solve the problem, then the problem was in the analyst's control. Therefore, only submit data from the extraction and analysis with surrogate spike recoveries within the control windows. This will be considered the initial analysis and will be reported as such on all data deliverables.

5.3.4 If the reextraction and reanalysis of the sample do not solve the problem (i.e., surrogate recoveries are outside the control windows for both analyses), then submit the surrogate spike recovery data and the sample data from both analyses. Distinguish between the initial analysis and the reanalysis on all data deliverables.

5.4 Documentation

The laboratory will report surrogate recovery data for the following.

5.4.1 Method blank analysis

5.4.2 Sample analysis

5.4.3 Matrix spike/matrix spike duplicate analysis

5.4.4 All sample reanalyses that substantiate a matrix effect

6. Matrix spike/matrix spike duplicate (MS/MSD) analysis

6.1 MS/MSD frequency of analysis

A matrix spike and matrix spike duplicate must be performed for each group of samples of a similar matrix, once each case of field samples has been received, or each 20 field samples in a case have been analyzed, whichever is more frequent.

6.2 Use the compounds listed in Attachment 10 to prepare matrix spiking solutions. Optional dilution steps must be accounted for when calculating the percent recovery of the matrix spike and matrix spike duplicate samples.

Note: Samples requiring optional dilutions and chosen as the MS/MSD samples must be analyzed at the same dilution as the original unspiked sample.

6.3 Individual component recoveries of the matrix spike are calculated using the following Equation.

$$\text{Matrix spike percent recovery} = \frac{SSR - SR}{SA} \times 100$$

Where:

SSR = Spike sample results.
SR = Sample result.
SA = Spike added from spiking mix.

6.4 Relative percent difference (RPD)

The analyst is required to calculate the RPD between the matrix spike and the matrix spike duplicate. RPDs for each component is calculated using the following Equation.

$$RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

Where:

RPD = Relative percent difference
D_1 = First sample value
D_2 = Second sample value (duplicate)

6.5 The matrix spike results (concentrations) for nonspiked, semivolatile TCL compounds are reported, and the matrix spike percent recoveries are summarized (Attachment 11).

APPARATUS:

- Soxhlet extractor, 47 mm i.d. x 170 mm length, Pyrex 3740 L.
- Drying column, 19 mm I.D. chromatographic column with coarse frit. Substitution of a small pad of Pyrex glass wool for the frit will prevent cross-contamination of sample extracts.

- Chromatographic column, glass, 1 cm i.d. x 25 cm length, with 250 mL reservoir, sintered glass frit, and Teflon stopcock
- Concentrator tube, Kuderna-Danish (K-D), 10 mL, graduated, Kontes K-570050-1025. The calibration must be checked at the volumes employed in the test. A ground glass stopper is used to prevent evaporation of extracts.
- Evaporative flask, KD, 500 mL, Kontes K-570001 0500. Attach the flask to the concentrator tube with springs.
- Snyder column, KD, three-ball macro, Kontes K503000 0121
- Snyder column, KD, two-ball micro, Kontes K569001 0219
- Beaker, 250 mL
- Vials, amber glass, 2-mL capacity, with Teflon-lined screw caps.
- Erlenmeyer flask, 500 mL, with ground glass joint 24/40.
- Silicon carbide boiling chips, 10/40 mesh. Heat the chips to 400°C for 30 minutes or soxhlet extract them with methylene chloride.
- Water bath, heated, with concentric ring cover, capable of temperature control (±2°C). The bath should be used in a hood.
- Balance, analytical, capable of accurately weighing ±0.0001 g
- Nitrogen evaporation device equipped with a water bath that can be maintained at 35 to 40°C, N-Evap, Organomation Associates, Inc.
- Gas chromatograph, analytical system complete with a temperature programmable gas chromatograph suitable for splitless injection and all required accessories including syringes, analytical columns, and gases
- Column, 30 m x 0.25 mm (or 0.32 mm), bonded-phase silicone-coated fused silica capillary column, J&W Scientific DB-5. A film thickness of 1.0-micron is recommended because of its larger capacity. A film thickness of 0.25-microns may be used.
- Glass chromatographic column for alumina, 6 mL, 150 mm x 8 mm i.d., column Kontes K-420155 or 5-mL serological pipettes plugged with a small piece of Pyrex glass wool or polyethylene porous disk, Kontes K-420162.
- Pyrex glass wool, prerinsed with appropriate solvents to ensure its cleanliness.

- Mass spectrometer, capable of scanning from 35 to 500 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum that meets all required criteria when 50 ng of decafluorotriphenylphosphine (DFTPP) is injected through the gas chromatograph inlet.

  Note: DFTPP criteria must be met before any sample extracts are analyzed.

- Data system: A computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and plot such ion abundances versus time or scan number. This type of plot is defined as an extracted ion current profile (EICP). Software must also be available that allows integrating the abundance in any EICP between specified time or scan number limits.

Note: Equivalent equipment may be substituted.

REAGENTS:

- Reagent water (Water in which an interferant is not observed at or above the method detection limit of each parameter of interest.)
- Sodium hydroxide solution, 10N. Dissolve 40 g of sodium hydroxide in reagent water and dilute it to 100 mL.
- Sodium thiosulfate, ACS, granular
- Sulfuric acid solution (1:1). Slowly add 50 mL of sulfuric acid, (specific gravity 1.84) to 50 mL of reagent water.
- Ethanol, 200 proof
- Acetone, hexane, methanol, and methylene chloride, pesticide quality
- 2% Ethanol/methylene chloride. Add 20 mL of ethanol to 980 mL of methylene chloride.
- Sodium sulfate, ACS, powdered, Baker anhydrous powder, Catalog No. 73898. Purify the sodium sulfate by heating it at 400°C for 4 hours in a shallow tray, cool it in a desiccator, and store it in a glass bottle.
- Florisil, pesticide residue grade. Activate in an oven at 120°C overnight.
- Surrogate standards, phenol-d_6, 2,4,6-tribromophenol, 2-fluorophenol, nitrobenzene-d_5, terphenyl-d_{14}, and 2-fluorobiphenyl. These are added to all samples and calibration solutions.

- Surrogate standard spiking solution, containing the base/neutral compounds at a concentration of 100 µg/mL and the acid compounds at 200 µg/mL. Store the spiking solutions at 4°C ±2°C in Teflon-sealed containers. The solutions should be checked frequently for stability. These solutions must be replaced after at least 12 months, if comparison with quality control check samples indicates a problem.

- Base/neutral/acid (BNA) Matrix standard spiking solution consisting of:

Base/Neutrals	Acids
1,2,4-trichlorobenzene	pentachlorophenol
acenaphthene	phenol
2,4-dinitrotoluene	2-chlorophenol
pyrene	4-chloro-3-methylphenol
N-nitroso-di-n-propylamine	4-nitrophenol
1,4-dichlorobenzene	

- Prepare the matrix spiking solution to contain each of the above compounds in methanol, with the base/neutrals at 100 µg/1.0 mL and the acid compounds at 200 µg/mL.

- Internal standards, 1,4-dichlorobenzene-d_4, naphthalene-d_8, acenaphthene-d_{10}, phenanthrene-d_{10}, chrysene-d_{12}, perylene-d_{12}. An internal standard solution can be prepared by dissolving 200 mg of each compound in 50 mL of methylene chloride. It may be necessary to use 5 to 10% benzene or toluene in this solution and a few minutes of ultrasonic mixing in order to dissolve all the constituents. The resulting solution will contain each standard at a concentration of 4,000 ng/µL. A 10-µL portion of this solution should be added to each 1 mL of sample extract (5 µL to 0.5 mL). This will yield a concentration of 40 ng/µL of each constituent.

- Calibration standards prepared at 20, 50, 80, 120, and 160 ng/µL. Each calibration standard should contain each compound of interest and each surrogate standard.

  Note: Great care must be taken to maintain the integrity of all standard solutions. Store all standard solutions at -10° to -20°C in screw-cap amber bottles with Teflon-liners. Fresh standards should be prepared at least every 12 months. The continuing calibration standard should be prepared weekly and stored at 4°C ±2°C.

Note: Equivalent reagents may be substituted.

PROCEDURE:

1. Sample storage and holding times

   1.1 Sample storage

   The samples must be protected from light and frozen at -20°C from the time of receipt until extraction and analysis.

   1.2 Holding times

   There are no holding times established for fish tissue samples.

2. Sample extraction

   2.1 Attach Soxhlet extractors (47 mm i.d. x 170 mm length) to 500-mL Erlenmeyer flasks with ground glass joints.

   2.2 Add two plugs of glass wool to each extractor, one to cover the bottom to prevent the sample from entering the solvent return arm and the other to cover the top of the sample.

   2.3 Add 300 mL of glass distilled methylene chloride to the Erlenmeyer flask, along with about five boiling chips. Attach the Erlenmeyer to the Soxhlet extractor.

   2.4 Attach the extractors to the condensers in the fume hood.

   2.5 Adjust the temperature so the extractors cycle at a rate of 12 to 15 cycles per hour.

   2.6 Allow the extractors to rinse for 4 hours, then shut off the heaters and allow them to cool.

   2.7 Remove the condensers and drain all the solvent remaining in the extractors into the Erlenmeyer flask.

   2.8 Discard the solvent and rinse the Erlenmeyer flask with two additional 10-mL portions of methylene chloride.

   2.9 The extractors are now ready for the samples.

   2.10 Weigh 30 g of sodium sulfate or the control matrix into a 250-mL beaker; this will represent the method blank.

   2.11 Weigh two 30 portions of the matrix spike and matrix spike duplicates into 250-mL beakers; these will be used for the matrix spikes.

2.12 Weigh 30-g samples into 250-mL beakers.

2.13 Add 60 g of anhydrous sodium sulfate to each beaker and mix the samples thoroughly, using a stainless steel spatula. More sodium sulfate may be necessary (when a sufficient amount has been added, the sample will appear granular).

2.14 Place the beakers in a fume hood and let them dry for at least 3 hours, stirring occasionally.

2.15 Add 2.0 mL of surrogate spiking solution to each beaker.

2.16 Add 2.0 mL of the matrix spiking solution to each 30-g portion of the sample that was selected as the matrix spike and matrix spike duplicate.

2.17 Remove the glass wool plug from the Soxhlet extractors that have been prerinsed.

2.18 Transfer the entire sample to the extractor and place the glass wool plug on top.

Note: The sample level in the extractor should not exceed the top of the solvent return arm; this will keep the entire sample immersed in solvent during the extraction process.

2.19 Add 150 mL of methylene chloride to the mixing beaker, swirl it and add the solvent to the respective Erlenmeyer flask along with about five boiling chips.

2.20 Attach the Soxhlet extractor to the Erlenmeyer flask.

2.21 Add 200 mL of glass-distilled methylene chloride to each Soxhlet extractor.

2.22 Attach the condensers and set the temperature so that the extractors cycle at a rate of 12 to 15 cycles per hour.

2.23 Let the extractors cycle for 16 hours.

2.24 After 16 hours, shut off the heating elements and allow the samples to cool.

2.25 Drain all of the solvent remaining in the extractor into the Erlenmeyer flask.

2.26 Rinse the extractor with about 50 mL of methylene chloride and drain it into the collection Erlenmeyer flask.

2.27 Assemble a K-D concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K-D if equivalency is demonstrated for all compounds listed in this method.

2.28 Pour the extracts through powder funnels containing Whatman #4 filter paper and collect the extracts in the K-D concentrators. Rinse the Erlenmeyer flasks and powder funnels with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

2.29 Add one or two clean boiling chips to the evaporative flasks and attach a three-ball Snyder column. Prewet the Snyder columns by adding about 1 mL of methylene chloride to the top. Place the K-D apparatus on a hot water bath (80° to 90°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatuses and the water temperature, as required, to complete the concentration in 10 to 15 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 5 to 10 mL, remove the K-D apparatus. Allow it to drain and cool for at least 10 minutes.

2.30 Adjust the volume to 10 mL using methylene chloride.

3. Lipid determination

3.1 Set up a log book page to include the following information.

3.1.1 Pan identification number

3.1.2 Sample identification number

3.1.3 Initial pan weight

3.1.4 Final pan and sample weight

3.1.5 Weight of lipids

3.1.6 Percent lipids

3.2 Weigh the aluminum pans on an analytical balance and record weights to four decimal places in a log book.

3.3 Using a disposal pipette, aliquot 1.0 mL of the sample extracts into the pans. record the HWI number in the log book with the corresponding pan identification and weight.

3.4 Place the pans in a hood and loosely cover them with foil (leave the foil loose enough to allow adequate air flow over the samples).

3.5 Let the samples dry overnight (approximately 15 hours).

3.6 Remove the samples from the hood, weigh them on an analytical balance, and record the weight to four decimal places in the log book.

3.7 Determine the weight of lipid as follows.

Weight of lipids = Final pan and sample weight - Initial pan weight.

3.8 The weight of lipid in the 1.0-mL aliquot is used to determine the number of loops required for the GPC cleanup. The maximum weight of lipid that should be loaded on the column at one time is 1.0 g. This means that the maximum extract concentration of lipids would be equivalent to 0.2 g/mL (e.g., 0.2 g/mL using a 5.0-mL sample loop would introduce 1.0 g of lipid to the column).

3.9 Record the weight of lipid in the log book.

3.10 Calculate the percent lipid as follows.

$$\% \text{ Lipid} = \frac{\text{Total weight of lipid in the (10 mL) extract}}{\text{Total weight of sample extracted}} \times 100\%$$

3.11 Record the percent lipid information in the log book.

4. GPC cleanup of extracts

4.1 The remaining 9-mL extract should have the lipids removed by gel permeation chromatography (OP-6004-36).

4.2 Optional: In cases where there is only sufficient sample to be extracted once, remove 4 mL of the 9 mL of remaining extract and save it in case a problem develops during the processing. Dilute the other 5 mL to 10 mL in methylene chloride before loading it onto the GPC.

5. Florisil cleanup of extracts

5.1 Quantitatively transfer the GPC eluent into a 500-mL K-D flask with a 10-mL concentrator tube attached.

5.2 Add a boiling chip and attach a three-ball Snyder column to the K-D apparatus.

5.3 Place the K-D apparatus on a hot water bath and evaporate the methylene chloride down to about 10 mL. Do not allow the sample to go dry!

5.4 Remove the K-D apparatus from the hot water bath. Remove the Snyder column and add about 60 mL of hexane and another boiling chip to the K-D flask. Mix the solvents by tilting the K-D apparatus.

5.5 Put the Snyder column back on the K-D flask, put the K-D apparatus back on the water bath and evaporate the solvent down to about 2 mL.

5.6 Allow the K-D flask to sit and cool for at least 10 minutes.

5.7 Remove the Snyder column and rinse the K-D flask with about 2 mL of hexane.

5.8 Remove the K-D flask from the concentrator tube.

5.9 Place the concentrator tube under a gentle stream of nitrogen and take the sample to a volume of 5 mL.

5.10 The sample may now be cleaned using Florisil.

5.10.1 Place 7 g of the activated Florisil in the column and tap gently to settle the adsorbent bed.

5.10.2 Wash the Florisil by allowing approximately 30 mL MeCl2/EtOH to pass through the column. Close the stopcock when the solvent level is approximately 1 cm above the adsorbent bed. Discard the eluant.

5.10.3 Use the entire sample extract from GPC. Reduce the sample extract volume to about 2 mL and transfer it quantitatively to the column with three 2-mL portions of MeCl2. Place a receiving flask under the column, open the stopcock and allow the extract to percolate through the column. As the solvent level approaches the level of the adsorbent bed, rinse down the sides of the column with about 2 mL of MeCl2 and allow this rinsing to pass onto the column until the solvent level approaches the top of the Florisil bed. Elute the column with 150 mL of the 2% ethanol in methylene chloride solvent mixture.

5.11 Concentrate the eluant to about 4 mL in a KD tube. Concentrate the extract further to just under 1 mL with a gentle stream of nitrogen gas.

5.12 Add 10 µL of BNA internal standard mixture.

5.13 Adjust the final volume to 1.0 mL with methylene chloride.

5.14 Transfer the extract to a GC auto-injection vial.

5.15 Analyze by GC/MS.

6. Calibration

6.1 Each GC/MS system must have the hardware tuned to meet the criteria listed in Attachment 3 for a 50-ng injection of DFTPP. No sample analyses can begin until all these criteria are met. These criteria must be demonstrated each 12-hour shift. DFTPP has to be injected to meet this criterion. Post-acquisition manipulation of abundances is not acceptable.

6.2 The internal standards should permit most components of interest in a chromatogram to have retention times of 0.80 to 1.20 relative to the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantification (Attachment 5). If interferences are noted, use the next most intense ion as the secondary ion (e.g., for 1,4-dichlorobenzene-d_4 use m/z 152 for quantification)

6.3 The internal standards are added to all calibration standards and all sample extracts just before analysis by GC/MS. A 10-µL aliquot of the internal standard solution should be added to a 1-mL aliquot of calibration standards.

6.4 Analyze 1 µL of each calibration standard and tabulate the area of the primary characteristic ion against concentration for each compound, including the surrogate compounds. Calculate RRFs for each compound using the following:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured.

A_{is} = Area of the characteristic ion for the specific internal standard from Attachment 5.

C_{is} = Concentration of the internal standard (ng/µL).

C^x = Concentration of the compound to be measured (ng/µL).

6.4.1 The average RRF should be calculated for all compounds. A system performance check must be made before this calibration curve is used. Four compounds (the system performance check compounds) are checked for a minimum average RRF. These compounds (the SPCCs) are N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol.

6.4.2 A % RSD is calculated for 11 compounds labeled the CCCs on Form VI SV and in Attachment 7. A maximum %RSD is also specified for these compounds. These criteria must be met for the calibration curve to be valid.

6.5 A check of the calibration curve must be performed once every 12 hours during analysis. These criteria are described in detail 5 in the Quality Assurance section. The minimum RRF for the system performance check compounds must checked. If this criterion is met, the RRFs of all compounds are calculated. A percent difference of the daily (12-hour) RRF compared with the average RRF from the initial curve is calculated. A maximum percent difference of 30% is allowed for each compound flagged as "CCC." Only after both these criteria are met can sample analysis begin.

6.6 Internal standard responses and retention times in all standards must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12-hour) calibration standard, the chromatographic system must be inspected for malfunctions and corrections must be made as required. The EICP of the internal standards must be monitored and evaluated for each standard. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction and corrections must be made as appropriate. When corrections are made, the samples analyzed while the system was malfunctioning must be reanalyzed.

7. GCMS analysis

7.1 The following instrumental parameters are required for all performance tests and for all sample analyses.

Electron energy - 70 volts (nominal)
Mass range - 35 to 500 amu
Scan time - not to exceed 1 second/scan

7.2 Internal standard solution (10 µL) is added to each sample extract. Analyze the extract by GC/MS using a bonded-phase silicone-coated fused silica capillary column. The recommended GC operating conditions to be used are as follows:

Initial column temperature hold: 40°C for 4 minutes
Column temperature program: 40° to 270°C at 10°C/minute
Final column temperature hold: 270°C for 10 minutes
Injector temperature: 250° to 300°C
Transfer line temperature: 250° to 300°C
Source temperature: According to manufacturer's specifications
Injector: Grob-type, splitless
Sample volume: 1 µL
Carrier gas: Helium at 30 cm^3/second

8. Qualitative analysis

8.1 The compounds listed in the TCL will be identified by an analyst who is competent in the interpretation of mass spectra by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the identifications: elution of the sample component at the GC relative retention time as the standard component, and correspondence of the sample component and standard component mass spectra.

8.1.1 For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within ±0.06 RRT units of the RRT of the standard component. For reference, the standard must be run on the same shift as the sample. If co-elution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

8.1.2 For comparison of standard and sample component mass spectra, mass spectra obtained on the analyst's GC/MS are required. Once obtained, these standard spectra may be used for identification purposes, only if the GC/MS meets the DFTPP daily tuning requirements. These standard spectra may be obtained from the run used to obtain reference RRTs.

8.1.3 The requirements for qualitative verification by comparison of mass spectra are as follows:

8.1.3.1 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.

8.1.3.2 The relative intensities of ions specified in Step 5.1.3.1 must agree within ±20% between the standard and sample spectra. (e.g., for an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 30% and 70%).

8.1.3.3 Ions greater than 10% in the sample spectrum, but not present in the standard spectrum, must be considered and accounted for by the analyst making the comparison. The verification process should favor false positives. All compounds meeting the identification criteria must be reported with their spectra. For all compounds below the method detection limit (MDL) report the actual value followed by "J" (e.g., "3J").

8.1.4 If a compound cannot be verified by all the criteria in Step 5.1.3, but the identification is correct in the technical judgement of the mass spectral interpretation specialist, then the contractor will report that identifications and proceed with quantification (Calculations section).

8.2 A library search will be executed for non-TCL sample components for the purpose of tentative identification. For this purpose, the 1985 release of the National Bureau of Standards (NBS) Mass Spectral Library (or a more recent release), containing 42,261 spectra, will be used.

8.2.1 Up to 20 nonsurrogate organic compounds of greatest apparent concentration not listed for the combined BNA fraction will tentatively be identified via a forward search of the NBS mass spectral library. (Substances with responses less than 10% of the nearest internal standard are not required to be searched in this fashion.) Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

8.2.2 Guidelines for making identification.

8.2.2.1 Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

8.2.2.2 The relative intensities of the major ions should agree within ±20% (e.g., for an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 30 and 70%).

8.2.2.3 Molecular ions present in reference spectrum should be present in sample spectrum.

8.2.2.4 Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or presence of coeluting compounds.

8.2.2.5 Ions present in the reference spectrum but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting compounds.

8.2.3 If in the technical judgement of the mass spectral interpretation specialist no valid, tentative identification can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (e.g., unknown phthalate, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If probable molecular weights can be distinguished, include them.

CALCULATIONS:

1. Identified TCL components will be quantified by the internal standard method. The internal standard used will be the one with a retention time nearest that of a given analyte. The EICP area of characteristic ions of analytes listed in Attachments 4 and 5 are used.

   Internal standard responses and retention times in all samples must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12-hour) calibration standard, the chromatographic system must be inspected for malfunctions and corrections must be made as required. The EICP of the internal standards must be monitored and evaluated for each sample, blank, matrix spike, and matrix spike duplicate. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction and corrections must be made as appropriate. When corrections are made, the samples analyzed while the system was malfunctioning must be reanalyzed.

2. The RRF from the daily standard analysis is used to calculate the concentration in the sample. Secondary ions may be used if interferences are present. The area of a secondary ion cannot be substituted for the area of a primary ion unless an RRF is calculated using the secondary ion. When TCL compounds are below quantitation limits, but the spectra meet the identification criteria, report the concentration with a "J."

   Calculate the concentration in the sample using the RRF, as determined in Step 4.3 of the Procedure section and the following equation.

$$\text{Concentration } \mu g/kg = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RRF)(W_s)(V_i)(D)}$$

   A_x = Area of the characteristic ion for the compound to be measured

   A_{is} = Area of the characteristic ion for the internal standard

   I_s = Amount of internal standard injected in nanograms (ng)

   W_s = Weight of sample extracted (grams)

   V_i = Volume of extract injected (μL)

   V_t = Volume of total extract (the volume is 2000 μL if GPC cleanup is used).

$$D = \frac{100 - \%\ \text{moisture}}{100}$$

3. An estimated concentration for tentatively identified non-TCL components will be quantified by the internal standard method. For quantification, the nearest internal standard free of interferences will be used.

   The formula for calculating concentrations is the same as that in Step 20. Total area counts (or peak heights) from the total ion chromatograms are to be used for both the compound to be measured and the internal standard. An RRF of 1 is to be assumed. The value from this quantitation will be qualified as estimated. This estimated concentration should be calculated for all tentatively identified compounds, as well as those identified as unknowns.

4. Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within the limits and report it.

   4.1 If the recovery is not within the limits (i.e., if two surrogates from either base/neutral or acid fractions are out of limits, or if recovery of any one surrogate in either fraction is below 10%), the following steps are required.

4.1.1 Check to be sure there are no errors in calculations, surrogate solutions, and internal standards.

4.1.2 Check instrument performance.

4.1.3 Reanalyze the sample if none of the above reveal a problem.

4.2 If none of the steps above solve the problem, then reextract and reanalyze the sample. If the reextraction and reanalysis of the sample solves the problem, then the problem was within the laboratory's control. Therefore, only submit data from the analysis with surrogate spike recoveries within the control windows. This will be considered the initial analysis and will be reported as such.

4.3 If the reextraction and reanalysis of the samples does not solve the problem (i.e., the surrogate recoveries are outside the control limits for both analyses), then submit the surrogate spike recovery data and the sample analysis data from analysis of both sample extracts. Distinguish between the initial analysis and the reanalysis on all data deliverables.

4.4 If the sample with surrogate recoveries outside the limits is the sample used for the matrix spike and matrix spike duplicate, and the surrogate recoveries of the matrix spike and the matrix spike duplicates show the same pattern (i.e., outside the limits), then the sample, matrix spike, and matrix spike duplicate do not require reanalysis. Document in the narrative the similarity in surrogate recoveries.

TARGET COMPOUND LIST

CAS No.	Compound	Target Detection Limits (µg/kg)
108-95-2	Phenol	660
111-44-4	bis (2-Chloroethyl) Ether	660
95-57-8	2-Chlorophenol	660
541-73-1	1,3-Dichlorobenzene	660
106-46-7	1,4-Dichlorobenzene	660
100-51-6	Benzyl Alcohol	660
95-50-1	1,2-Dichlorobenzene	660
95-48-1	2-Methylphenol	660
39638-32-9	bis(2-Chloroisopropyl)Ether	660
106-44-5	4-Methylphenol	660
621-64-7	N-Nitroso-Di-n-Propylamine	660
67-72-1	Hexachloroethane	660
98-95-3	Nitrobenzene	660
78-59-1	Isophorone	660
88-75-5	2-Nitrophenol	660
105-67-9	2,4-Dimethylphenol	660
65-85-0	Benzoic Acid	3,200
111-91-1	bis(2-Chloroethoxy)Methane	660
120-83-2	2,4-Dichlorophenol	660
120-82-1	1,2,4-Trichlorobenzene	660
91-20-3	Naphthalene	660
106-47-8	4-Chloroaniline	660
87-68-3	Hexachlorobutadiene	660
59-50-7	4-Chloro-3-methylphenol	660
91-57-6	2-Methylnaphthalene	660
77-47-4	Hexachlorocyclopentadiene	660
88-06-2	2,4,6-Trichlorophenol	660
95-95-4	2,4,5-Trichlorophenol	3,200
91-58-7	2-Chloronaphthalene	660
88-74-4	2-Nitroaniline	3,200
131-11-3	Dimethyl Phthalate	660
208-96-8	Acenaphthylene	660
606-20-2	2,6-Dinitrotoluene	660
99-09-2	3-Nitroaniline	3,200
83-32-9	Acenaphthene	660
51-28-5	2,4-Dinitrophenol	3,200
100-02-7	4-Nitrophenol	3,200
132-64-9	Dibenzofuran	660
121-14-2	2,4-Dinitrotoluene	660
84-66-2	Diethylphthalate	660
7005-72-3	4-Chlorophenyl-Phenylether	660
86-73-7	Fluorene	660
100-10-6	4-Nitroaniline	3,200
534-52-1	4,6-Dinitro-2-Methylphenol	3,200
86-30-6	N-Nitrosodiphenylamine (1)	660

TARGET COMPOUND LIST

CAS No.	Compound	Target Detection Limits (µg/kg)
101-55-3	4-Bromophenyl-Phenylether	660
118-74-1	Hexachlorobenzene	660
87-86-5	Pentachlorophenol	3,200
85-01-8	Phenanthrene	660
120-12-7	Anthracene	660
84-74-2	Di-n-butylphthalate	660
206-44-0	Fluoranthene	660
129-00-0	Pyrene	660
85-68-7	Butylbenzylphthalate	660
91-94-1	3,3'-Dichlorobenzidine	1,300
56-55-3	Benzo(a)anthracene	660
218-01-9	Chrysene	660
117-81-7	bis(2-Ethylhexyl)phthalate	660
117-84-0	Di-n-octylphthalate	660
205-99-2	Benzo(b)fluoranthene	660
207-08-9	Benzo(k)fluoranthene	660
50-32-8	Benzo(a)pyrene	660
193-39-5	Indeno(1,2,3-cd)Pyrene	660
53-70-3	Dibenz(a,h)Anthracene	660
191-24-2	Benzo(g,h,i)perylene	660

SEMIVOLATILE HAZARDOUS SUBSTANCE LIST COMPOUNDS
FISH QUALITY CONTROL SUMMARY

Compound	Spike Added (ppm)	Determinant Number of Samples	Average % Recovered	Standard Deviation
Phenol	1.67	15	70.5	13.97
bi(2-Chloroethyl)ether	1.67	14	55.6	11.15
2-Chlorophenol	1.67	16	60.6	13.83
1,3-Dichlorobenzene	1.67	16	35.8	18.54
1,4-Dichlorobenzene	1.67	16	36.3	17.77
Benzyl Alcohol	1.67	14	57.7	23.09
1,2-Dichlorobenzene	1.67	16	40.2	19.65
2-Methylphenol	1.67	16	63.2	20.35
bis(2-Chloroisopropyl)ether	1.67	16	55.1	19.04
4-Methylphenol	1.67	16	69.1	12.37
N-Nitroso-di-n-propylamine	1.67	14	68.9	21.94
Hexachloroethane	1.67	16	35.1	19.31
Nitrobenzene	1.67	16	61.9	20.32
Isophorone	1.67	14	75.4	19.96
2-Nitrophenol	1.67	15	59.9	18.81
2,4-Dimethylphenol	1.67	14	67.4	20.98
Benzoic Acid	8.33	16	0.0	0.00
bis(2-Chloroethoxy)methane	1.67	16	73.4	18.48
2,4-Dichlorophenol	1.67	16	72.6	13.04
1,2,4-Trichlorobenzene	1.67	15	60.4	19.87
Naphthalene	1.67	16	65.7	18.21
4-Chloroaniline	1.67	16	20.9	15.30
Hexachlorobutadiene	1.67	15	52.0	17.13
4-Chloro-3-methylphenol	1.67	16	72.7	15.17
2-Methylnaphthalene	1.67	16	71.3	14.36
Hexachlorocyclopentadiene	1.67	15	3.1	5.71
2,4,6-Trichlorophenol	1.67	16	82.2	17.46
2,4,5-Trichlorophenol	8.33	16	74.6	14.22
2-Chloronaphthalene	1.67	14	77.1	15.14
2-Nitroaniline	8.33	15	84.7	15.57
Dimethyl Phthalate	1.67	14	90.9	21.83
Acenaphthylene	1.67	16	87.1	19.27
3-Nitroaniline	8.33	15	55.7	24.17
Acenaphthene	1.67	16	85.5	17.59
2,4-Dinitrophenol	8.33	16	6.1	16.09
4-Nitrophenol	8.33	14	72.4	28.58
Dibenzofuran	1.67	16	86.2	17.44
2,4-Dinitrotoluene	1.67	16	85.1	20.32
2,6-Dinitrotoluene	1.67	16	86.8	18.14
Diethylphthalate	1.67	14	89.6	27.49
4-Chlorophenyl-phenylether	1.67	16	83.0	17.39
Fluorene	1.67	16	83.8	17.17
4-Nitroaniline	8.33	16	29.0	20.54
4,6-Dinitro-2-methylphenol	8.33	15	21.5	28.12

SEMIVOLATILE HAZARDOUS SUBSTANCE LIST COMPOUNDS
FISH QUALITY CONTROL SUMMARY

Compound	Spike Added (ppm)	Determinant Number of Samples	Average % Recovered	Standard Deviation
N-Nitrosodiphenylamine	1.67	16	75.4	19.38
4-Bromophenyl-phenylether	1.67	16	86.7	17.53
Hexachlorobenzene	1.67	15	93.3	17.86
Pentachlorophenol	8.33	14	75.4	29.38
Phenanthrene	1.67	14	94.1	18.20
Anthracene	1.67	16	86.5	19.24
Di-n-butylphthalate	1.67	13	102.2	30.33
Fluoranthene	1.67	14	100.1	21.28
Pyrene	1.67	14	79.2	19.82
Butylbenzyphthalate	1.67	14	97.0	29.70
3,3-Dichlorobenzidine	3.33	15	7.8	15.13
Benzo(a)anthracene	1.67	14	84.1	20.05
bis(2-Ethylhexyl)phthalate	1.67	13	88.7	39.12
Chrysene	1.67	16	86.8	20.77
Di-n-octyl Phthalate	1.67	13	65.9	33.01
Benzo(b)Fluoranthene	1.67	14	85.2	23.90
Benzo(k)Fluoranthene	1.67	14	90.6	21.89
Benzo(a)Purene	1.67	15	77.2	19.84
Indeno(1,2,3-cd)pyrene	1.67	13	74.4	38.30
Dibenzo(a,h)anthracene	1.67	13	81.8	36.12
Benzo(g,h,i)perylene	1.67	13.	63.9	32.40

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	30.0% to 60.0% of mass 198
68	Less than 2.0% of mass 69
70	Less than 2.0% of mass 69
127	40.0% to 60.0% of mass 198
197	Less than 1.0% of mass 198
198	Base peak, 100% relative abundance
199	5.0% to 9.0% of mass 198
275	10.0% to 30.0% of mass 198
365	Greater than 1.00% of mass 198
441	Present but less than mass 443
442	Greater than 40.0% of mass 198
443	17.0% to 23.0% of mass 442

CHARACTERISTIC IONS FOR SEMIVOLATILE TCL COMPOUNDS

Parameter	Primary Ion	Secondary Ion(s)
Phenol	94	65, 66
bis(-2-chloroethyl)ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene	146	148, 113
Benzyl alcohol	108	79, 77
1,2-Dichlorobenzene	146	148,113
2-Methylphenol	108	107
bis(-2-chloroisoprophyl)ether	45	77, 79
4-Methylphenol	108	107
N-Nitroso-di-propylamine	70	42, 101, 130
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	65, 109
2,4-Dimethylphenol	107	121, 122
Benzoic acid	122	105, 77
bis(-2-chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223, 227
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,200
2-Chloronaphthalene	162	164, 127
2-Nitroaniline	65	92, 138
Dimethyl phthalate	163	194, 164
Acenaphthylene	152	151, 153
3-Nitroaniline	138	108, 92
Acenaphthene	153	152, 154
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	109	139, 65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 182
2,6-Dinitrotoluene	165	89, 121
Diethylphthalate	149	177, 150
4-Chlorophenyl-phenylether	204	206, 141
Fluorene	166	165, 167
4-Nitroaniline	138	92, 108
4,6-Dinitro-2-Methylphenol	198	182, 77
N-Nitrosodiphenylamine	169	168, 167
4-Bromophenyl-phenylether	248	250, 141
Hexachlorobenzene	284	142, 249

CHARACTERISTIC IONS FOR SEMIVOLATILE TCL COMPOUNDS

Parameter	Primary Ion	Secondary Ion(s)
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Anthracene	178	179, 176
Di-n-Butylphthalate	149	150, 104
Fluoranthene	202	101, 100
Pyrene	202	101, 100
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Benzo(a)anthracene	228	229, 226
bis(2-ethylhexyl)phthalate	149	167, 279
Chrysene	228	226, 229
Di-n-octyl phthalate	149	-
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 125
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenz(a, h)anthracene	278	139, 279
Benzo(g, h, i)perylene	276	138, 277

MP-HZBS-MA
ATTACHMENT 5

CHARACTERISTIC IONS FOR SURROGATES AND INTERNAL STANDARDS FOR SEMIVOLATILE COMPOUNDS

Parameter	Primary Ion	Secondary Ion(s)
Surrogates		
Phenol-d_5	99	42, 71
2-Fluorophenol	112	64
2,4,6-Tribromophenol	330	332, 141
d-5 Nitrobenzene	82	128, 54
2-Fluorobiphenyl	172	171
Terphenyl	244	122, 212
Internal Standards		
1,4-Dichlorobenzene-d_4	152	115
Naphthalene-d_8	136	68
Acenaphthene-d_{10}	164	162, 160
Phenanthrene-d_{10}	188	94, 80
Chrysene-d_{10}	240	120, 236
Perylene-d_{12}	264	260, 265

Semi-Volatile Internal Standards with Corresponding TCL Analytes Assigned for Quantitation

1,4-Dichlorobenzene-d	Naphthalene-d	Acenaphthene-d	Phenanthrene-d	Chrysene-d	Perylene-d
Phenol	Nitrobenzene	Hexachlorocyclo-pentadiene	4,6-Dinitro-2-methylphenol	Pyrene	Di-n-octyl phthalate
bis(2-chloroethyl) ether	Isophorone	2,4,6-Trichloro-phenol	N-nitrosodi-phenylamine	Butylbenzyl phthalate	Benzo(b)fluor-anthene
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichloro-phenol	1,2-Diphenylhy-drazine	3,3-Dichloro-benzidine	Benzo(k)fluor-anthene
1,3-Dichlorobenzene	2,4-Dimethyl-phenol	2-Chloronaphthalene	4-Bromophenyl phenyl ether	Benzo(a)-anthracene	Benzo(a)pyrene
1,4-Dichlorobenzene	Benzoic acid	2-Nitroaniline	Hexachloro-benzene	bis(2-ethyl-hexyl)phthalate	Indeno(1,2,3-c pyrene
Benzyl alcohol	bis(2-Chloro-ethoxy)methane	Dimethyl phthalate	Pentachloro-phenol	Chrysene	Dibenz(a, h) anthracene
1,2-Dichlorobenzene	2,4-Dichloro-phenol	Acenaphthylene	Phenanthrene	Terphenyl-d_{14} (surr)	Benzo(g, h, i) perylene
2-Methylphenol	1,2,4-Trichloro-benzene	3-Nitroaniline	Anthracene		
bis(2-Chloroiso-propyl)ether	Napthalene	Acenaphthene	Di-n-butyl phthalate		
4-Methylphenol	4-Chloroaniline	2,4-Dinitrophenol	Fluoranthene		
N-nitroso-Di-n-propylamine	Hexachloro-butadiene	4-Nitrophenol			
Hexachloroethane	4-Chloro-3-methylphenol	Dibenzofuran			
2-Fluorophenol (surr)	2-Methylnaphth-alene	2,4-Dinitrotoluene			
Phenol-d_6 (surr)	Nitrobenzene-d_5 (surr)	2,6-Dinitrotoluene			
		Diethyl phthalate			
		4-Chlorophenyl-phenyl ether			
		Fluorene			
		4-Nitroaniline			
		2-Fluorobiphenyl (Surr)			
		2,4,6-Tribromophenol			

Calibration Check Compounds

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-Methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitroso-di-n-phenylamine	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

Continuing calibration: 50 ng

MP-HZBS-MA
ATTACHMENT 8

Surrogate Spiking Compounds

Compound	Amount in Sample Fraction	(µg)
Nitrobenzene-d_5	BNA	200
2-Fluorobiphenyl	BNA	200
p-Terphenyl-d_{14}	BNA	200
Phenol-d_5	BNA	400
2-Fluorophenol	BNA	400
2,4,6-Tribromophenol	BNA	400

MP-HZBS-MA
ATTACHMENT 9

Required Surrogate Spike Recovery Limits

Fraction	Surrogate Compound	%
BNA	Nitrobenzene-d_5	23-120
BNA	2-Fluorobiphenyl	30-115
BNA	p-Terphenyl-d_{14}	18-137
BNA	Phenol-d_5	24-113
BNA	2-Fluorophenol	25-121
BNA	2,4,6-Tribromophenol	19-122

MP-HZBS-MA
ATTACHMENT 10

Matrix Spiking Solutions

Base/Neutrals	Spike Amount (µg)	Acids	Spike Amount (µg)
1,2,4-Trichlorobenzene	200	Pentachlorophenol	400
Acenaphthene	200	Phenol	400
2,4-Dinitrotoluene	200	2-Chlorophenol	400
Pyrene	200	4-Chloro-3-Methylphenol	400
1,4-Dichlorobenzene	200	4-Nitrophenol	400

MP-HZBS-MA
ATTACHMENT 11

Matrix Spike Recovery Limits

Fraction	Matrix Spike Compound	(%)	%RSD
BN	1,2,4-Trichlorobenzene	10-120	33
BN	Acenaphthene	29-145	23
BN	2,4-Dinitrotoluene	24-146	24
BN	Pyrene	20-139	25
BN	N-Nitroso-Di-n-Propylamine	10-135	32
BN	1,4-Dichlorobenzene	10-90	49
Acid	Pentachlorophenol	10-164	39
Acid	Phenol	28-112	20
Acid	2-Chlorophenol	19-102	23
Acid	4-Chloro-3-Methylphenol	27-118	21
Acid	4-Nitrophenol	10-158	40